THE SYNTHESIS, MAGNETIC STUDIES, AND SINGLE CRYSTAL X-RAY DIFFRACTION STRUCTURES OF POLYNUCLEAR COPPER (II) OXIME IMINES OF 2,3-BUTANEDIONE

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THE SYNTHESIS, MAGNETIC STUDIES, AND
SINGLE CRYSTAL X-RAY DIFFRACTION STRUCTURES OF POLYNUCLEAR
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TABLE OF CONTENTS

LIST OF TABLES

LIST OF TABLES (CONTINUED)

LIST OF ILLUSTRATIONS

SUMMARY

Polynuclear complexes of copper(II) with several oxime imines of 2,3-butanedione have been synthesized, characterized by elemental analysis and infrared spectra, analyzed magnetically at various temperatures, and examined by single crystal x-ray diffraction to determine their crystal and molecular structures. Copper-copper distances in all cases are large enough to minimize direct metal exchange and insure that any spin coupling is due to superexchange.

Bis{perchlorato[2-(2-hydroxyethyl)imino-3-oximo-butanato] aquo copper(II)} was found to have magnetic susceptibilities of 64×10^{-6}, 54×10^{-6}, and 28×10^{-6} cgsu per copper atom at 77, 195, and 298^{o}K, respectively. The centrosymmetric dimer consists of two copper atoms with tetragonally distorted octahedral coordination. The axial sites are occupied by a perchlorate oxygen and a water oxygen while the in-plane sites are occupied by two nitrogens (from the oxime and the imine), an oxygen from the hydroxyethyl group, and an oxygen from the oxime of the ligand on the other metal in the dimeric unit. A planar six-membered copper-nitrogen-oxygen ring formed by this dimeric structure affords superexchange by a σ or π mechanism.

Bis{2[-(α-pyridyl)ethyl]imino-3-oximo-butanato acetonitrile copper(II) perchlorate} forms dimeric complex cations having a two-fold axis of symmetry. The asymmetric unit contains a copper atom with square-pyramidal coordination. The axial site is occupied by the nitrogen atom of the acetonitrile; and the other four sites are occupied by three nitrogen

atoms (oxime, imine, and pyridine) and one oxygen atom from the oxime on the ligand of the other copper in the dimer. The dimer contains a half-boat shaped six-membered copper-nitrogen-oxygen ring which has a two-fold axis perpendicular to the center of the roughly planar ring. The desolvated complex and the dihydrate have been synthesized but have slightly different magnetic properties. The magnetic susceptibilities of the nitrile, dihydrate, and desolvated complex are 180.8×10^{-6}, 353.9×10^{-6}, and 363×10^{-6} cgsu, respectively, at 297±1°K, whereas all three complexes drop down to less than 80×10^{-6} cgsu at 93°K.

Bis{perchlorato-bis 2-(2-aminoethyl)-imino-3-oximo butanato dicopper(II) perchlorate} forms tetrameric units with magnetic susceptibilities of 37×10^{-6}, 164×10^{-6}, 630×10^{-6}, and 815×10^{-6} cgsu per copper atom at 65, 90, 159, and 295°K, respectively. The centrosymmetric tetramer consists of two dimers joined by a four-membered ring made up of a copper atom and an oxime oxygen atom from each dimer. The two copper atoms in the dimeric unit are joined by oxime bridging groups into a six-membered ring with a half-boat conformation. Each copper atom has square-pyramidal coordination with basal positions occupied by nitrogen atoms (oxime, imine, and amine) and an oxygen atom of the bridging oxime; the axial position of one copper atom is occupied by the oxime oxygen of the adjacent dimer which forms the four-membered ring and the axial position of the second copper atom is occupied by a perchlorate oxygen atom. Superexchange probably occurs within the dimeric units through the six-membered rings rather than through the four-membered ring.

The compound (2,10-dioximo-3,9-dimethyl-4,8-diaza-undecanato) hemimethanol copper(II) perchlorate forms infinite zig-zag chains of copper

complexes with tetragonally distorted octahedral coordination. The nitrogen atoms (two oxime and two imine) at one ligand occupy the four in-plane coordination positions and one axial position is occupied by the oxime oxygen of an adjacent unit, forming the chain. The other axial position is occupied by the oxygen of the methanol of solvation for half of the units and is vacant for the remaining units. The minimum copper-copper distance is 5.129(4) Å and there is no magnetic coupling in this copper(II) complex.

CHAPTER I

INTRODUCTION

Although most copper(II) coordination complexes exhibit the normal paramagnetic properties expected for a d^9 system, several polynuclear complexes exhibit anomolous magnetic behavior. The magnetic behavior of these complexes has been attributed to spin coupling through a variety of bridging groups, but certainly the most extensively studied complexes involve oxygen as a single atom bridge. Several structure determinations have revealed oxime groups serving as bridges between copper(II) atoms and several copper(II) oxime complexes have been reported to have unusual magnetic properties. The purpose of this work is to systematically study, through synthesis, investigation of magnetic properties, and single crystal x-ray diffraction structure determinations, polynuclear copper(II) complexes with oxime imines of 2,3-butanedione. These oxime imine complexes offer a wide variation in magnetic properties with relatively small changes in the ligand environment of the copper atom.

Most copper(II) complexes, because of the one unpaired electron in the d^9 configuration, are paramagnetic and are attracted into a magnetic field. For a simple paramagnetic substance this attraction is positive, inversely related to absolute temperature, and varies directly with the strength of the magnetic field. Compounds containing unpaired electrons may exhibit more complex magnetic behavior such as ferromagnetism or antiferromagnetism. In both of these cases, the substance shows normal paramagnetic behavior above a certain temperature. Below a certain temperature,

called the Curie temperature, the magnetic susceptibility of a ferromagnetic substance increases at a greater rate with decreasing temperature. The magnetic susceptibility of an antiferromagnetic material decreases with decreasing temperature at a certain temperature called the Néel temperature. Figure 1 illustrates the relationship of the molar susceptibility (corrected for diamagnetism), χ_M^{corr}, to absolute temperature in paramagnetic, antiferromagnetic, and ferromagnetic substances.

Although antiferromagnetic behavior can result from direct interaction, or bonding, of metal atoms, most examples of both antiferromagnetic and ferromagnetic behavior involve superexchange through bridging groups. Figure 2 illustrates the orbital diagrams and corresponding energy level diagrams for various types of exchange coupling through a single-atom bridge. Oxygen-bridged copper(II) complexes have been studied extensively and show the effect of structural variation, such as chelate ring size, on the structures and magnetic properties of such complexes. The complexes of 2-(2-hydroxyethyl)imino-pentane-4-one and 2-(3-hydroxyproply)imino-pentane-4-one with copper(II)[1] are a weakly antiferromagnetic tetramer and a strongly antiferromagnetic dimer, respectively. For a series of hydroxy-bridged copper(II) dimers, the magnetic properties vary regularly from ferromagnetic to antiferromagnetic as the Cu-O-Cu angle is increased.[2]

Although the magnetic behavior of single-atom bridged systems is reasonably well understood, there have been few investigations of systems with two-atom bridges. Although the structures of relatively few such complexes have been reported, it is possible for the oxime group to function as a two-atom (N-O) bridge. In fact, because of the presence of one electron pair on nitrogen and three electron pairs on oxygen in the deprotonated

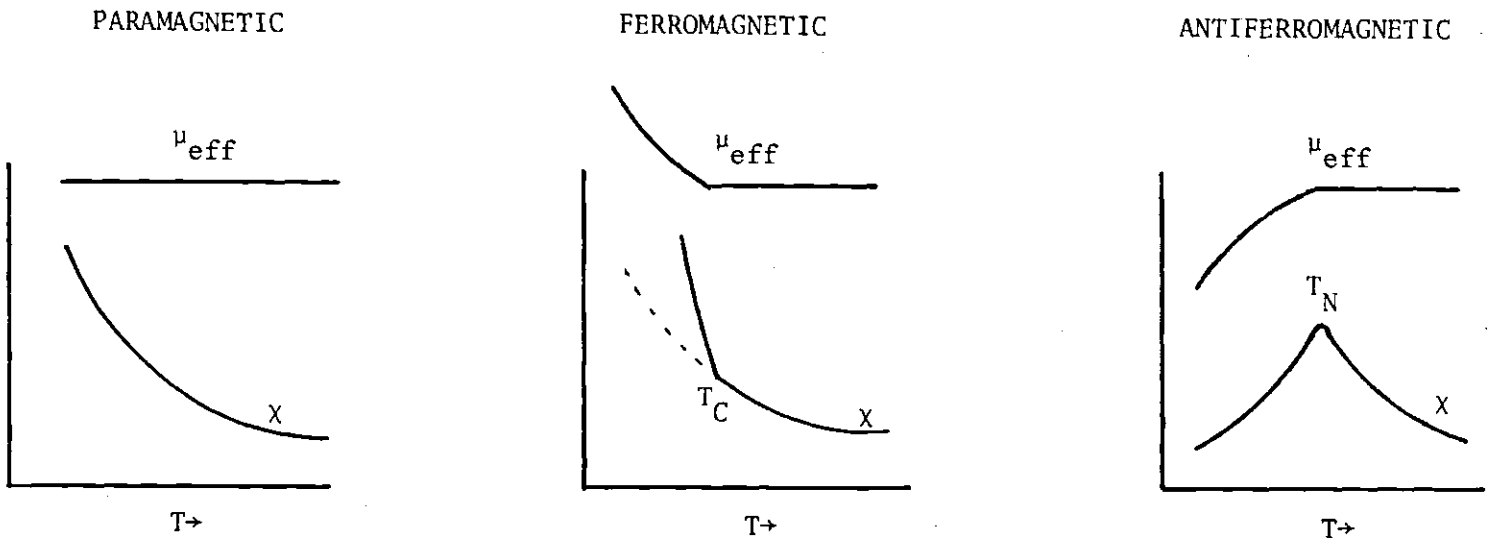


Figure 1. Comparison of χ and μ_{eff} versus $T_{°K}$ in Paramagnetic, Ferromagnetic, and Antiferromagnetic Substances.

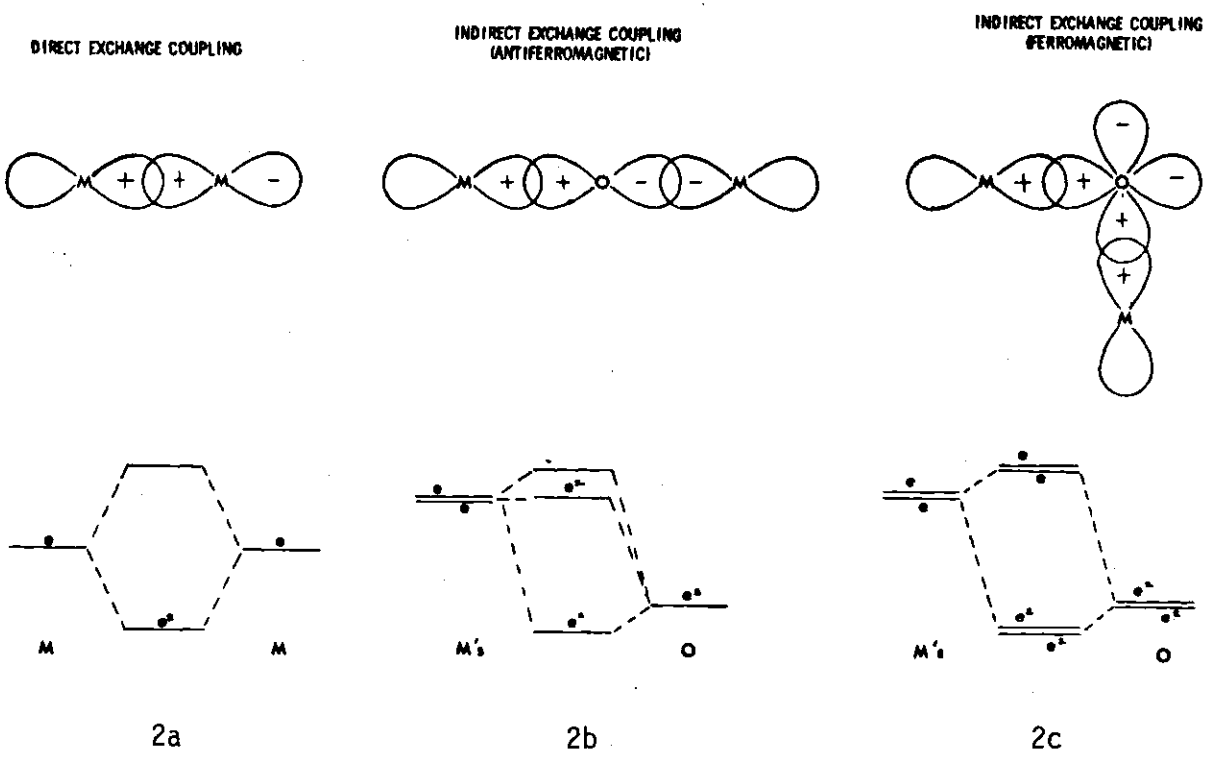
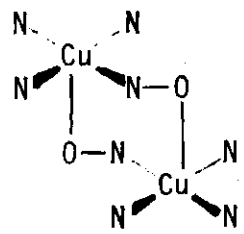


Figure 2. Orbital Diagrams for Exchange Coupling and Orbital Energy Splitting.

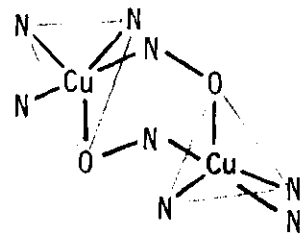
oxime group, such groups may potentially serve as either a two-atom bridge or a one-atom bridge or both.

The earliest reported oxime complex was the red nickel(II) complex of dimethylglyoxime reported by L. Tsugaeff.[3] This report led to widespread investigations to determine the exact structure of this new complex, and finally Godycki and Rundle[4] reported the single crystal x-ray diffraction structure of bis(dimethylglyoximato)nickel(II). Since 1905 a great deal or research on oxime coordination complexes,[5] mostly with vicinal dioximes such as dimethylglyoxime, has been reported and a few of the crystal structures of these oxime complexes have been decided by single crystal x-ray diffraction. It is interesting to note that it was not the nickel(II) dimethylglyoxime complex, but bis(dimethylglyoximato)-copper(II) which was the first oxime coordination complex for which single crystal x-ray diffraction studies were reported.[6]

There have been four single crystal x-ray diffraction studies of oxime bridged polynuclear complexes, only one of which has a reduced magnetic moment. In bis(dimethylglyoximato)copper(II), $Cu(dmgH)_2$, dimeric units (Ā) have a chair conformation of copper-oxime oxygen-oxime nitrogen atoms in a six membered centrosymmetric ring with the two square pyramidal copper atoms separated by a distance of 3.84 Å.[7]

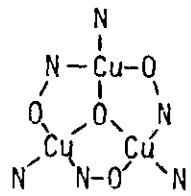


Ā



B

Bis(4,4,9,9-tetramethyl-5,8-diazadodecane-2,11-dione-2-oxime-μ-11-oximato)dicopper(II) bromide, Cu(4499)Br, forms a boat conformation (B) with the copper-oxime oxygen-oxime nitrogen atoms in a six-membered ring. The two trigonal bipyramidal copper atoms are related by a two-fold axis and separated by 3.949(4) Å.[8] A dimeric nickel oxime amine, bis[N-(6-amino-4-aza-3,3,6-trimethyl-2-heptylidenyl)hydroxanato] pentaaquo dinickel(II) chloride, abbreviated Ni(NAH)Cl·2.5H_2O, has a slightly distorted planar centrosymmetric nickel-oxime oxygen-oxime nitrogen six-membered ring with square planar coordination of the nickel atoms and a nickel-nickel distance of 3.631(2) Å.[9] The structure of the trimeric complex μ_3-hydroxo-tri-μ-(pyridine-2-carbaldehyde oximato)-μ_3-sulphato-tricopper(II)-16.3 water, $[Cu_3(pyal)_3OH]SO_4 \cdot 16.3H_2O$, with a magnetic moment of about 1.0 BM per copper atom, consists of three square pyramidal copper atoms forming five-membered copper-oxime oxygen-oxime nitrogen-copper-hydroxide oxygen rings (C) with copper-copper distances of 3.220(3) Å.[10]



C

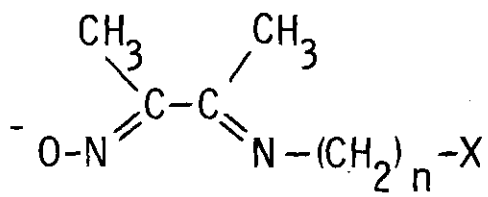
There have been several interesting polynuclear copper oxime complexes reported including three copper complexes which have low magnetic moments and for which the imine ligands are derived from 2,3-butanedione monoxime. (These ligands and others studied in this work can be abbreviated as LnX,

Figure 3.) In 1964 V. E. Uhlig and D. Schneider reported the synthesis of $Cu(L2py)ClO_4$ and found it to have a magnetic moment of 1.04 BM at 292°K.[11] A. V. Ablov and N. I. Belichuk[12] synthesized $Cu(L2a\ell)ClO_4 \cdot H_2O$ and reported a magnetic moment of 0.41 BM at ambient temperature. And in 1974 J. G. Mohanty, S. Baral, R. P. Singh, and A. Chakravorty[12] reported the synthesis of $[Cu_3(Let)_3O]ClO_4$ with a magnetic moment of 1.03 BM per copper atom at ambient temperature and the synthesis of $[Cu(L2dea)(ClO_4)]_2 \cdot H_2O$ with antiferromagnetic behavior and a magnetic moment of 0.69 BM at 273°K.

The antiferromagnetic behavior of these complexes suggests polynuclear structures. In $Cu(L2a\ell)(ClO_4) \cdot H_2O$ bridging may reasonably involve (1) the alcoholic oxygen as in the oxygen bridged structures, 2-(2-hydroxyethyl)imino-pentane-4-onato copper(II) and 2-(3-hydroxypropyl)imino-pentane-4-onato copper(II), (2) the oxime oxygen, or (3) the oxime group. The other copper(II) oxime imines with low magnetic moments have only the second and third possibilities involving the oxime atoms.

Identification of the bridging arrangement and geometry of these polynuclear complexes is important in the explanation of their magnetic properties and the difference in magnetic properties for these complexes and those previously observed to have oxime bridges. In order to understand the bridging possibilities of oxime groups, to relate the bridging possibilities to the magnetic properties, and to elucidate the exchange mechanism, a study of these complexes was initiated.

This work began by attempts to synthesize $Cu(L2a\ell)ClO_4 \cdot H_2O$, $Cu(L2pyr)ClO_4$, and $[Cu(Let)_3O](ClO_4)$ in crystals suitable for single crystal x-ray diffraction studies. $Cu(LOsc)(ClO_4)$, which was mentioned



Ln X

n will always be an integer

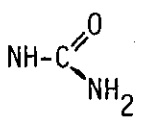
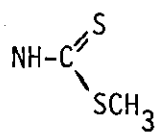
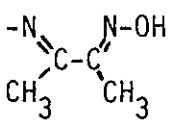
n	X	Symbol	n	X	Symbol
n=2,3	OH	Lnaℓ	0		LOsc
n-2,3	NH_2	Lnam			
2	α-pyridine	L2pyr	0		LOdts
2	$(C_2H_5)_2N$	L2dea			
1	CH_3	Let			
			3		L3LH

Figure 3. Abbreviations for Ligands and Complexes Described in this Thesis.

in the literature[14] with no magnetic data, was of interest and was included in the study. Several new complexes, $Cu(L3a\ell)(ClO_4)\cdot H_2O$, $Cu(L2am)(ClO_4)$, $Cu(L3am)(ClO_4)$, $Cu(L3LH)(ClO_4)\cdot \frac{1}{2}CH_3OH$, and Cu(LOdts)Br, were synthesized and studied.

The single crystal x-ray diffraction structures of $Cu(L2a\ell)(ClO_4)\cdot H_2O$ with a low magnetic moment at ambient temperatures, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$ and $Cu(L2am)(ClO_4)$ with intermediate magnetic moments at 296°K, 0.68 BM for $Cu(L2pyr)(ClO_4)\cdot CH_3CN$ and 1.23 BM for $Cu(L2am)(ClO_4)$, and $Cu(L3LH)(ClO_4)\cdot \frac{1}{2}CH_3OH$ with a normal (1.79 BM at 296°K) magnetic moment are reported in this thesis.

CHAPTER II

EXPERIMENTAL

Preparation of Complexes

$Cu(L2a\ell)(ClO_4)\cdot H_2O$

The method of Ablov, et al.[12] was used to synthesize $Cu(L2a\ell)(ClO_4)\cdot H_2O$. Dark green prismatic crystals were obtained by recrystallization from methanol. Anal. Calc'd for $Cu(C_6H_{11}N_2O_2)(ClO_4)\cdot H_2O$: C, 22.23; H, 4.04; N, 8.64. Found: C, 22.02; H, 3.69; N, 8.54. All elemental analyses were performed by Atlantic Microlab, Inc. in Atlanta, Georgia.

$Cu(L3a\ell)(ClO_4)\cdot H_2O$

Dark green crystals of $Cu(L3a\ell)(ClO_4)\cdot H_2O$ were obtained by employing the same method as for $Cu(L2a\ell)(ClO_4)\cdot H_2O$, substituting 3-amino-1-propanol for 2-aminoethanol. Anal. Calc'd for $Cu(C_7H_{13}N_2O_2)(ClO_4)\cdot H_{20}$: C, 24.86; H, 4.47; N, 8.28; Cℓ, 10.48. Found C, 25.05; H, 4.20; N, 8.44: Cℓ, 10.59.

$Cu(L2pyr)(ClO_4)$

The method of Uhlig and Schneider[11] was used to obtain green black needles of $Cu(L2pyr)(ClO_4)$. Anal. Calc'd for $Cu(C_{11}H_{14}N_3O)(ClO_4)$: C, 35.97; H, 3.84; N, 11.44; Cℓ, 9.65. Found: C, 35.92; H, 4.18; N, 11.18; Cℓ, 9.80. The solvates $Cu(L2pyr)(ClO_4)\cdot 2H_2O$ and $Cu(L2pyr)(ClO_4)\cdot CH_3CN$ were synthesized by crystallizing $Cu(L2pyr)(ClO_4)$ from water and acetonitrile respectively. The compound was recrystallized from water, the density was then determined by flotation with a mixture of carbon

tetrachloride and 1,2-dibromo-1,1-dichloroethane, and the volume of the unit cell calculated from the unit cell parameters determined by single crystal x-ray diffraction. Calculation of the molecular weight indicated the compound to be the dihydrate. The acetonitrile solvate was characterized by a complete single crystal x-ray diffraction structure.

Cu(L2am)(ClO_4)

The ligand (L2amH) was prepared by adding 50 mmoles of diacetyl monoxime in 25 ml of methanol to 50 mmoles of ethlenediamine in 25 ml of methanol and bringing this solution to a boil. When this mixture was added to 50 mmoles of cupric perchlorate hexahydrate in 50 ml of methanol and the mixture brought to a boil and filtered, green-black prisms of Cu(L2am)(ClO_4) precipitated. After recrystallization from methanol the following analysis was obtained. Anal. Calc'd for Cu($C_6H_{12}N_3O$)(ClO_4): C, 23.61; H, 3.96; N, 13.77. Found: C, 24.03; H, 4.04; N, 13.86.

Cu(L3am)(ClO_4)

By replacing ethylenediamine by 1,3-diaminopropane in the preparation of Cu(L2am)(ClO_4) a green leaf-like solid precipitated. When this material was recrystallized from cold ethanol, green-black prisms, which rapidly decomposed in air, formed. The green leaf-like solid was recrystallized from methanol. Anal. Calc'd for Cu($C_7H_{14}N_3O$)(ClO_4): C, 26.34; H, 4.42; N, 13.16. Found: C, 25.98; H, 4.30; N, 12.91.

Cu(LOsc)(ClO_4)

The method of Ablov and Belichuk[14] produced a pale green powder which was very slightly soluble in ethanol. This material was recrystallized from ethanol. Anal. Calc'd for Cu($C_5H_9N_4O_2$)(ClO_4)·H_2O: C, 17.75; H, 3.28. Found: C, 17.80; H, 3.21.

Cu(LOdts)Br

50 mmoles of S-methyldithiocarbozate[15] in 25 ml of ethanol and 50 mmoles of diacetyl monoxime in 25 ml of ethanol were boiled and left to evaporate. The resulting white solid was recrystallized from ethanol to yield the S-methyldithiocarbazone of diacetyl monoxime $C_6H_{11}N_3OS_2$ which decomposes at 195°C. Anal. Calc'd for $C_6H_{11}N_3OS_2$: C, 35.10; H, 5.40; N, 20.47. Found: C, 35.16; H, 5.42; N, 20.58. Ten mmoles of this ligand in 25 ml of methanol was added slowly to ten mmoles of cupric bromide in 25 ml of methanol. Bronze green leaflets precipitated. These leaflets were recrystallized from hot acetone to form brown green prisms. Anal. Calc'd for $Cu(C_6H_{10}N_3O)Br$: C, 20.72; H, 2.90; N, 12.08. Found: C, 20.96; H, 2.94; N, 12.17.

$Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$

Ten mmoles of 1,3-diaminopropane were added slowly to 20 mmoles of diacetyl monoxime in 25 ml of ethanol. This mixture was stirred and refluxed for one hour. To this hot solution was added slowly and with rapid stirring ten mmoles of cupric perchlorate hexahydrate. Evaporation of the methanol and cooling yielded wine-red prisms. Anal. Calc'd for $Cu(C_{11}H_{19}N_4O_2)(ClO_4)\cdot\frac{1}{2}CH_3OH$: C, 33.00; H, 5.03; N, 13.38; Cl, 8.48. Found: C, 32.48; H, 4.98; N, 13.79; Cl, 8.63.

Magnetic Studies

The magnetic susceptibilities of all complexes were obtained from data taken by the Faraday method. The system was calibrated with $Hg(Co(SCN)_4)$ and corrected for the diamagnetism of the quartz bucket. Values for the diamagnetic correction of each sample, χ_{dia}, were taken from Figgis and Lewis.[16]

Table 1 lists all complexes prepared in this study with name, diamagnetic correction of the sample, χ_{dia}; molar magnetic susceptibility per copper atom corrected for diamagnetism, χ_M^{corr}; and effective magnetic moment, μ_{eff}; calculated at 296, 195, and 93°K. For Cu(L2am)(ClO_4), Cu(L2pyr)(ClO_4)·CH_3CN, and Cu(L2pyr)(ClO_4)·$2H_2O$ magnetic data at various temperatures from 298° to 93°K are listed in Table 2.

Crystallographic Studies

General

All computations were carried out on a UNIVAC 1108 Exec 8 system or on a CDC Cyber 70/74 computer. Programs used include Carter's cell parameter and diffractometer setting angle program[17], Zalkin's FORDAP Fourier summation program,[18] Ibers' NUCLS5 modification of the Busing-Martin-Levy ORFLS full matrix least squares program,[19] the Busing-Martin-Levy ORFEE function and error program,[20] Johnson's ORTEP and ORTEPII plotting programs,[21] Stewart's ABSORB absorption correction program from the x-ray 72 system,[22] the software package for the Syntex $P2_1$ diffractometer, and various locally written programs.

In the structure factor calculations, the scattering factors for all atoms except hydrogen were taken from Cromer and Waber's tabulation;[23] Stewart's hydrogen atom scattering factors were used.[24] The scattering factors of the copper and chlorine atoms were corrected for the real and imaginary anomalous dispersion components using the dispersion factors tabulated by Cromer.[25] The agreement factors are defined in the usual way as:

$$R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$$

and

$$R_w = [\Sigma w(|F_o|-|F_c|)^2/\Sigma w(|F_o|)^2]^{\frac{1}{2}}$$

Table 1. Diamagnetic Corrections (χ_{dia}), Corrected Molar Magnetic Susceptibilities (χ_m^{corr}) at 298, 195, and 93°K, and Effective Magnetic Moment at 298, 195, and 93°K, for Complexes Prepared for this Thesis

	$\chi_{dia} \times 10^6$ cgsu	$\chi_m^{corr} \times 10^6$ cgsu			μ_{eff} BM		
		298	195	93	298	195	93
Cu(L2aℓ)(ClO_4)·H_2O	128	28	54	64	0.26	0.29	0.22
Cu(L3a)(ClO_4)·H_2O	140	51	-	34	0.35	-	0.16
Cu(L2am)(ClO_4)	108	816	762	164	1.38	1.09	0.34
Cu(L3am)(ClO_4)	133	1087	-	2410	1.62	-	1.34
Cu(L2pyr)(ClO_4)	154	363	154	-	0.93	0.49	-
Cu(L2pyr)(ClO_4)·$2H_2O$	177	354	193	73	0.92	0.55	0.22
Cu(L2pyr)(ClO_4)·CH_3CN	177	180	70	14	0.66	0.33	0.10
Cu(LO sc)ClO_4)·H_2O	102	77	-	-	0.43	-	-
Cu(LOdts)Br	148	1208	-	3987	1.70	-	1.68
Cu(L3LH)(ClO_4)·½CH_3OH	176	1301	-	4734	1.77	-	1.88

Table 2. Corrected Molar Magnetic Susceptibilities (χ_m^{corr}) and Effective Magnetic Moments (μ_{eff}) at Various Temperatures for the Formula Units of: $Cu(L2a\ell)(ClO_4)(H_2O)$[a]

$Cu(L2pyr)(ClO_4)\cdot CH_3CN$, and $Cu(L2pyr)(ClO_4)\cdot 2H_2O$

$Cu(L2a\ell)(ClO_4)(H_2O)$			$Cu(L2pyr)(ClO_4)\cdot CH_3CN$			$Cu(L2pyr)(ClO_4)\cdot 2H_2O$		
T°K	$\chi_m^{corr}\times 10^6$ cgsu	μ_{eff}	T°K	$\chi_m^{corr}\times 10^6$ cgsu	μ_{eff}	T°K	$\chi_m^{corr}\times 10^6$ cgsu	μ_{eff}
295	815.9	1.39	298	180.8	0.66	296	353.9	0.92
256	820.6	1.30	288	192.6	0.67	283	377.6	0.93
236	802.2	1.24	283	195.1	0.66	273	300.3	0.81
201	760.1	1.11	273	187.3	0.64	263	297.8	0.80
195	762.3	1.09	263	146.1	0.56	253	287.6	0.77
175	690.8	0.99	253	155.9	0.56	243	265.6	0.72
159	630.2	0.90	243	144.0	0.53	233	240.1	0.67
140	530.3	0.77	233	125.0	0.48	223	218.0	0.63
133	474.3	0.71	223	147.9	0.51	213	258.7	0.66
110	330.8	0.54	213	136.1	0.48	203	212.0	0.59
108	315.5	0.52	203	130.8	0.46	193	192.5	0.55
90	164.4	0.35	193	70.0	0.33	183	170.4	0.50
72	85.6	0.22	183	54.7	0.30	173	173.8	0.50
65	37.4	0.14	173	67.3	0.29	163	135.6	0.42
63	39.4	0.14	163	72.1	0.31	153	111.1	0.37
55	61.6	0.17	153	60.7	0.28	143	111.1	0.36
52	56.2	0.15	143	73.3	0.29	133	127.2	0.37
48	50.0	0.14	133	70.1	0.27	123	105.6	0.32
40	50.1	0.13	123	86.5	0.29	113	75.4	0.26
39	175.9	0.24	113	82.6	0.27	103	67.1	0.23
37	212.1	0.25	103	8.4	0.08	93	73.7	0.22
26	411.6	0.29	93	13.5	0.10			
24	453.9	0.30						

In all least squares refinements, the quantity minimized was $\Sigma (|F_o|-|F_c|)^2$. Numbers in parenthesis following other numbers indicate estimated standard deviations in the least significant digits.

The form of the anisotropic thermal ellipsoids is $\exp[-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}\ell^2 - \beta_{12}hk - \beta_{13}h\ell - \beta_{23}k\ell]$. All hydrogen atoms were refined isotropically with the thermal parameter B fixed at 5.0.

Cu(L2aℓ)(ClO_4)·H_2O

Precession photographs of the h0ℓ, h1ℓ, and hkk reciprocal lattice levels and the full diffractometer data set (vide infra) revealed Laue symmetry 2/m and the systematic absences 0k0 (k = 2n + 1) and h0ℓ (ℓ = 2n + 1), uniquely defining the space group as C_{2h}^5 - $P2_1/c$ (No. 14).[26] For data collection a well-formed, multifaceted crystal was mounted collinear with the (0kk) row of the reciprocal lattice on a glass fiber attached to a goniometer head. The crystal was placed on a Picker automated four-circle diffractometer and 14 medium-intensity reflections at a variety of 2θ values were accurately centered using Zr-filtered Mo Kα radiation (λ=.7093 Å). The setting angles from these reflections were used to refine cell parameters by a least-squares procedure; the cell parameters obtained are a = 7.868 (2), Å, b = 14.040 (7) Å, c = 11.401 (5) Å, and β= 112.73 (4)°. With the assumption of 4 formula units per unit cell, the calculated density, 1.85 g cm^{-3}, agrees with the density measured, 1.83 (2) g cm^{-3}, gy the flotation method in a carbon tetrachloride and 1,3-diiodopropane mixture. The intensities were measured with a scintillation counter mounted 26.5 cm from the crystal and a source-crystal distance of 21.0 cm; the intensities were collected by the θ-2θ scan technique with a takeoff angle of 1.9° and a scan rate of 1° min^{-1}. A symmetrical scan

of 2.0° was taken about the calculated position for each reflection out to a maximum 2θ angle of 50° (λ=0.70926 Å). Stationary-background counts of 20 sec. were taken at the beginning (bgd1) and at the end (bgd2) of the scan. Calibrated copper attenuators were used in the collection of data. The threshold point was set so that the counting rate would not exceed 10,000 counts/sec. The pulse height analyzer was set for approximately a 90% window centered on the Mo Kα peak. Three axial reflections (400, 060, 004) and a null reflection (090) were monitored after every 150 reflections as a check on crystal and instrument stability. An isotropic linear decrease of 8% in the intensities of standards was observed during data collection and appropriate corrections were applied. A total of 2076 unique reflections were collected in a full quadrant of reciprocal space with $2\theta \leq 50°$. Corrected intensities (CI) were obtained by subtracting 3 times the actual measured background from the total integrated peak count (CT)

$$CI = CT - 3(bgd1 + bgd2)$$

The factor of 3 in the calculation arises from the peak scan time's being 3 times as long as the background counting time. The corrected intensities were assigned standard deviations according to the formula

$$\sigma(I) = [CT + 0.25(t_c/t_b)^2(bgd1 + bgd2 + 9.0) + (pl)^2 + 4.5]^{\frac{1}{2}}$$

where t_c is the scan time, t_b is the counting time of each background (either bgd1 or bgd2), and the scalar quantities 4.5 and 9.0 account for truncation of the last counting digit by the Picker scalar. A total of 1579 reflections were accepted as statistically above background on the

basis that $\sigma(I)/CI$ be less than 0.333 with p set at 0.03. Lorentz and polarization corrections were applied in the usual way. Atomic coordinates for copper and chlorine, two oxygen atoms, and one nitrogen atom were deduced from a three-dimensional Patterson synthesis and refined to give the initial residuals, R = 0.288 and R_w = 0.340. The remaining oxygen, nitrogen, and carbon atoms were located by means of subsequent difference Fourier syntheses and least-squares refinements. Using a weighting scheme based on the variances ($w = 4I/\sigma(I)^2$) and employing isotropic temperature factors for all nonhydrogen atoms, a refinement reduced R to 0.109 and R_w to 0.139. When anisotropic temperature factors were refined for all nonhydrogen atoms, R = 0.090 and R_w = 0.092. The 12 principal faces of the crystal were identified as follows (distance from center of crystal to the face in mm given in parentheses): {100} (0.16), {010}(0.20),{001}(0.23), {111}(0.15), {111}(0.12), {011}(0.29). Absorption corrections were calculated by the gaussian quadrature method; corrections on F^2 ranged between 1.50 and 2.01. Refinement with the absorption-corrected data set, after deletion of several reflections having erroneous scans due to instrumental malfunctions, reduced R to 0.068 and R_w to 0.074. All hydrogen atoms were located on a difference Fourier synthesis and their coordinates were refined with temperature factors fixed at the isotropic value of the corresponding carbon or oxygen atom from the cycle before conversion to anisotropic. Two hydrogen atoms, H1C5 and H1C1, failed to refine properly and were fixed in calculated positions (C-H = 0.95 Å, H-C-H = 109.5°). The final refinements reduced R to 0.057 and R_w to 0.056. In the last cycle, the error in an observation of unit weight was 1.1 and the maximum parameter shifts were

Table 3. Individual Positional and Thermal Parameters

for $Cu(L2a\ell)(ClO_4)\cdot H_2O$

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{11}	β_{23}
Cu	-0.1206(1)	-0.0048(1)	0.1048(1)	0.0117(1)	0.0036(1)	0.0058(1)	0.0017(1)	0.0038(1)	0.0012(1)
Cℓ	0.2471(2)	0.1455(1)	0.3483(1)	0.0148(3)	0.0050(1)	0.0080(1)	-0.0006(1)	0.0055(2)	-0.0013(1)
O1	0.1824(6)	-0.0803(3)	0.0315(4)	0.0158(8)	0.0045(2)	0.0078(4)	0.0028(3)	0.0063(5)	0.0024(2)
O2	-0.3195(7)	0.0528(4)	0.1563(5)	0.0169(10)	0.0059(3)	0.0087(5)	0.0031(4)	0.0057(6)	0.0017(3)
O3	0.3385(8)	0.1255(4)	0.0333(5)	0.0197(11)	0.0050(3)	0.0081(5)	0.0018(4)	0.0037(6)	0.0010(3)
O4	0.1770(16)	0.1289(9)	0.2190(8)	0.0643(36)	0.0168(9)	0.0142(9)	0.0015(15)	0.0133(15)	0.0020(8)
O5	0.1010(14)	0.1760(7)	0.3775(11)	0.0485(26)	0.0159(8)	0.0288(15)	0.0114(12)	0.0280(17)	0.0068(9)
O6	0.3158(13)	0.0577(6)	0.4046(10)	0.0488(27)	0.0089(5)	0.0266(14)	0.0087(10)	0.0113(15)	0.0049(7)
O7	0.3847(16)	0.2117(9)	0.3864(16)	0.0473(30)	0.0143(8)	0.0482(27)	-0.0151(14)	0.0173(23)	-0.0083(13)
C1	0.3194(9)	-0.2027(4)	0.2338(7)	0.0156(12)	0.0040(3)	0.0087(6)	0.0029(5)	0.0042(7)	0.0026(4)
C2	0.1604(7)	-0.1351(3)	0.2089(5)	0.0097(9)	0.0025(2)	0.0049(4)	0.0002(4)	0.0019(5)	0.0004(3)
C3	0.0603(8)	-0.1296(4)	0.2955(5)	0.0125(11)	0.0037(3)	0.0061(5)	-0.0007(5)	0.0020(6)	0.0006(3)
C4	0.1303(12)	-0.1818(6)	0.4170(7)	0.0229(16)	0.0066(4)	0.0073(7)	0.0026(8)	0.0064(9)	0.0027(4)
C5	-0.1909(12)	-0.0502(7)	0.3293(7)	0.0245(17)	0.0095(6)	0.0076(7)	0.0057(8)	0.0087(9)	0.0031(5)
C6	-0.3642(11)	-0.0076(7)	0.2411(8)	0.0234(16)	0.0085(5)	0.0118(8)	0.0050(9)	0.0111(10)	0.0030(6)
N1	0.0984(6)	-0.0789(3)	0.1123(4)	0.0095(8)	0.0029(2)	0.0055(4)	-0.0001(3)	0.0028(5)	0.0004(2)
N2	-0.0771(7)	-0.0751(4)	0.2589(5)	0.0143(10)	0.0050(3)	0.0059(4)	0.0010(4)	0.0058(5)	0.0008(3)

Atom	x	y	z	B,Å^2	Atom	x	y	z	B,Å^2
H1C1	0.270	-0.260	0.220	3.5	H2C4	0.256(12)	-0.149(5)	0.467(7)	4.1
H2C2	0.362(10)	-0.209(5)	0.164(7)	3.5	H3C4	0.155(12)	0.232(6)	0.403(8)	4.1
H3C1	0.428(10)	-0.193(5)	0.328(7)	3.5	H1C5	-0.204	-0.101	0.378	4.5
H1O2	-0.388(11)	0.069(8)	0.116(8)	3.2	H2C5	-0.138(11)	0.014(5)	0.390(8)	4.5
H1O3	0.345(12)	-0.165(6)	-0.005(9)	3.6	H1C6	-0.477(11)	-0.077(6)	0.176(8)	4.6
H2O3	-0.301(12)	-0.133(6)	-0.077(8)	3.6	H2C6	-0.447(12)	0.029(5)	0.275(8)	4.6
H1C4	0.048(11)	-0.169(5)	0.481(7)	4.1					

Table 4. Selected Interatomic Distances (Å) and Angles (deg)[a] of $Cu(L2a\ell)(ClO_4)\cdot H_2O$

Distances			
Cu-Cu'	3.581(3)	Cℓ-O5	1.381(8)
Cu-O1'	1.870(4)	Cℓ-O6	1.399(8)
Cu-O2	2.038(5)	Cℓ-O7	1.365(9)
Cu-O3	2.492(6)	C1-H1C1	0.88 (10)
Cu-O4	2.890(12)	C1-H2C1	0.98 (8)
Cu-N1	1.987(5)	C1-H3C1	1.10 (7)
Cu-N2	1.928(5)	C4-H1C4	1.16 (9)
C1-C2	1.508(8)	C4-H2C4	1.05 (8)
C2-C3	1.484(8)	C4-H3C4	0.77 (9)
C3-C4	1.474(9)	C5-H1C5	0.93 (10)
C5-C6	1.474(11)	C5-H2C5	1.11 (8)
C2-N1	1.289(7)	C6-H1C6	1.24 (8)
C3-N2	1.257(7)	C6-H2C6	1.02 (9)
C5-N2	1.456(9)	O2-H1O2	0.60 (8)
C6-O2	1.428(10)	O3-H1O3	0.65 (9)
N1-O1	1.325(6)	O3-H2O3	0.68 (9)
Cℓ-O4	1.381(9)		

Angles			
N1-Cu-o1'	108.3(2)	C1-O4-Cu	120.8(7)
N1-Cu-N2	81.1(2)	C2-C1-H1C1	105 (4)
N2-Cu-O2	80.0(2)	C2-C1-H2C1	115 (4)
O2-Cu-O1'	90.2(2)	C2-C1-H3C1	113 (4)
Cu-N1-O1	129.4(3)	H1C1-C1-H2C1	92 (4)
N1-o1-Cu'	121.9(3)	H2C1-C1-H3C1	115 (6)
Cu'-Cu-O3	90.9(2)	H1C1-C1-H3C1	114 (4)
Cu'-Cu-O4	74.4(2)	C3-C4-H1C4	114 (4)
Cu-N1-C2	117.3(4)	C3-C4-H2C4	103 (4)
N1-C2-C3	114.4(5)	C3-C4-H3C4	107 (6)
C3-N2-Cu	116.2(4)	H1C4-C4-H2C4	103 (6)
Cu-N2-C5	116.6(4)	H2C4-C4-H3C4	105 (8)
N2-C5-C6	109.0(6)	H1C4-C4-H3C4	122 (8)
C5-C6-O2	107.4(6)	N2-C5-H1C5	112 (5)
C6-O2-Cu	112.1(4)	N2-C5-H2C5	112 (5)
C1-C2-N1	124.1(5)	C6-C5-H1C5	115 (5)
C2-C3-C4	119.8(5)	C6-C5-H2C5	97 (4)
C1-C2-C3	121.5(5)	H1C5-C5-H2C5	111 (4)
N2-C3-C4	125.3(6)	C5-C6-H1C6	103 (4)
C3-N2-C5	126.5(5)	C5-C6-H2C6	120 (5)

[a]Primed atoms are related by the inversion center at the origin to the atoms listed in Table 3.

Table 4 (Continued). Selected Interatomic Distances (Å) and Angles (deg) for $Cu(L2a\ell)(ClO_4)\cdot H_2O$

Angles			
O1-N1-C2	117.3(4)	O2-C6-H1C6	106 (4)
O4-Cℓ-O5	106.4(7)	O2-C6-H2C6	109 (5)
O4-Cℓ-O6	105.3(7)	H1C6-C6-H2C6	111 (6)
O5-Cℓ-O7	113.3(9)	C6-O2-H1O2	111 (9)
O5-Cℓ-O6	111.9(6)	H1O3-O3-H2O3	112(11)
O5-Cℓ-O7	110.2(8)	H1O3-O3-Cu	115 (8)
O6-Cℓ-O7	109.6(8)	H2O3-O3-Cu	101 (7)

Table 5. Least Squares Planes within the Molecule

$Cu(L2a1)(ClO_4)\cdot H_2O$[a,b]

Atom	Dev, Å	Atom	Dev, Å
(a) Plane of the Four Atoms Strongly Coordinated to Copper Equation: 0.44452x + 0.69685y + 0.56285z - 0.02125 = 0			
O1'	0.001	N1	-0.003
O2	-0.002	N2	0.003
(b) Plane of the Eight Donor Atoms from the L Groups of the Dimer Equation: -0.45001x - 0.69736y - 0.55783z = 0			
O1	0.018	O1'	0.018
O2	0.006	O2'	0.006
N1	-0.013	N1'	0.013
N2	-0.000	N2'	0.000
Cu	0.066	Cu'	-0.066
C1	-0.056	C1'	0.056
C2	-0.058	C2'	0.058
C3	-0.095	C3'	0.095
C4	-0.305	C4'	0.305
C5	-0.114	C5'	0.114
C6	0.425	C6'	-0.425
(c) Plane of the Six-Membered Ring Containing Two Copper Atoms Equation: -0.43401x - 0.69429y - 0.57411z = 0			
Cu	0.025	Cu'	-0.025
N1	0.032	N1'	-0.032
O1	-0.030	O1'	0.030
(d) Plane of the Iminooximobutane Fragment Equation: 0.44952x + 0.76925y + 0.45408z + 33861 = 0			
C1	-0.066	N1	0.150
C2	0.032	N2	-0.019
C3	-0.019	O1	0.154
C4	0.005	Cu	0.205

[a]Direction cosines of the plane refer to the orthogonal axis system a, b, c*.

[b]All atoms weighted at unity.

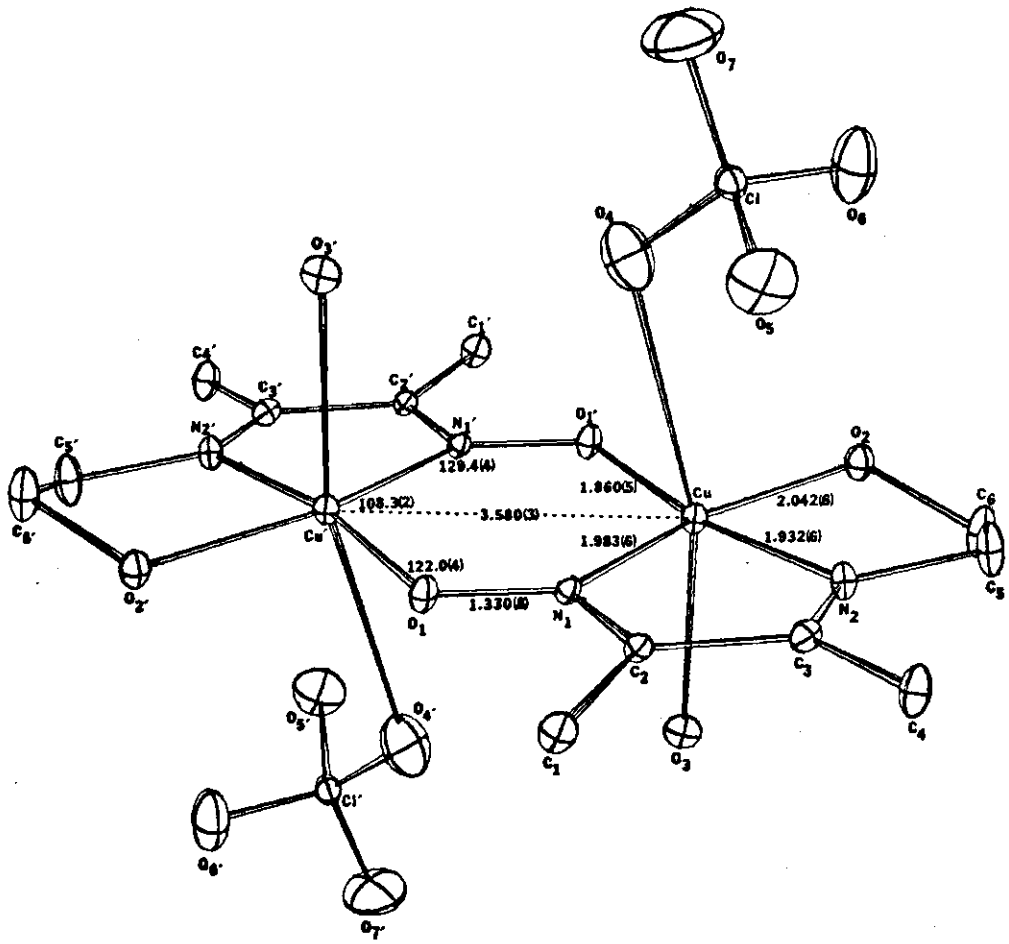
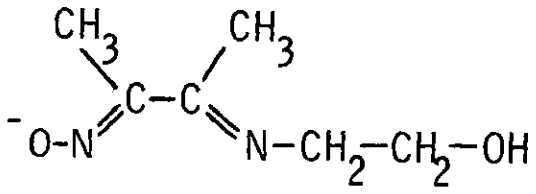


Figure 4. Dimeric Molecular Unit of $Cu(L2al)(ClO_4)\cdot H_2O$.

0.56σ(y coordinate for H3C4) and 0.39σ(y coordinate for C4). The only features greater than 0.50 e/Å^3 on a final difference Fourier map were a few small bumps of magnitude 0.52-0.84 e/Å^3 in the vicinity of the perchlorate group; a typical height for the perchlorate oxygens which were refined in this structure was about 4.5 e/Å^3.

Final atomic positional and thermal parameters are listed in Table 3; final observed and calculated structure factor amplitudes are listed in the Appendix. Selected interatomic distances and angles are tabulated in Table 4; selected least-squares planes are summarized in Table 5. The dimeric molecular unit is illustrated in Figure 4.

The four formula units of $Cu(L2a\ell)(ClO_4)\cdot H_2O$ in the unit cell form dimeric units about inversion centers at the origin and at (0, ½, ½); the dimeric unit is illustrated in Figure 4. Within the formula unit, the ligand



L2aℓ

is coordinated to the copper to form (1) a five-membered chelate ring involving the alcohol oxygen and the imine nitrogen and (2) a five-membered chelate ring involving the imine and oxime nitrogens. The fourth position in the coordination plane of the copper is occupied by the oxime oxygen of the second ligand of the dimer, resulting in bridging oxime groups; these four donor atoms are nearly coplanar, the maximum deviation from the plane being 0.003 Å, and the copper is 0.074 Å from the plane.

The bond distances and angles about copper, Table 5, are typical for complexes with five-membered chelate rings.[1,27,28] As is often true with copper(II) complexes, there are additional groups in axial positions; in this case there is a weakly bound water molecule (Cu-O3=2.492(6) Å) and an even more weakly bound perchlorate group (Cu-O4 = 2.89(2) Å completing the tetragonal coordination. The copper is slightly displaced from the basal plane toward the water molecule.

Charge-balance considerations dictate that one proton has been lost from the ligand in forming the complex. Although either the alcohol or oxime proton could be ionized, we feel that the oxime hydrogen has been ionized for the following reasons: (1) the oxime hydrogen should be more acidic than the alcohol proton, (2) the anionic portion of the ligand might be anticipated in the bridging moiety and the oxime does function in the bridge with appropriate geometry, (3) the region in the electron density map near the oxime group is quite flat whereas a good peak was located at a reasonable distance and direction from the alcohol oxygen atom, and (4) the alcohol hydrogen so located was successfully refined.

The most unusual feature of this structure is the bridging arrangement. A six-membered centrosymmetric ring is formed by two copper atoms and two oxime (NO) groups. Although not required by symmetry, all six atoms of the ring lie very close to the least-squares plane through the six atoms, the maximum deviations being for the nitrogen atoms (0.029 Å). The Cu-O1 (1.870(4) Å) and Cu-N1 (1.987(5) Å) distances indicate a strongly coordinated bridging oxime group and the Cu-O1'-N1' (121.9(3)°) and Cu-N1-O1 (129.4(3)° angles indicate essentially sp^2 hybridization for the oxime atoms.

A water molecule occupies an axial position although its distance from the copper (Cu-O3 = 2.492(6) Å) indicates weak coordination; the O3-Cu-basal donor angles range from 90.8(2) to 93.7(2)°. The perchlorate group appears to be ordered and occupies the sixth octahedral position. The geometries at oxygen (Cu-O4-Cl = 120.8(7)°) and at copper (O4-Cu-basal donor angles range from 77.4(3) to 101.1(3)° suggest a definite interaction despite the length (2.89(1) Å) of the Cu-O4 vector. The Cl-O stretching frequency is split into symmetric components at 1065 and 1132 cm^{-1}, a splitting that is normal for monodentate perchlorate coordination.[29]

The six-atom 2-imino-3-oximobutane fragment of L2aℓ adopts a configuration which is almost planar (maximum deviation from the least-squares plane is 0.15 Å for N1). The C2-C3 distance of 1.484(8) Å and the C2-N1 and C3-N2 distances of 1.289(7) and 1.257(7) Å, respectively, agree well with the corresponding distances in dimethylglyoxime structures[7] and indicate the absence of cyclic delocalization.

The ethanolimine portion of L2aℓ adopts a twist configuration similar to that found in many ethylenediamine chelate rings.[30] Thus, C5 and C6 lie 0.114 and 0.425 Å, respectively, above and below the least-squares plane through the eight donor atoms from the two ligands of the dimer. No unusual inter- or intramolecular contacts, other than those noted above, were observed.

$\underline{Cu(L2pyr)(ClO_4)\cdot CH_3CN}$

A blue black crystal with approximate dimensions 0.4 x 0.8 x 0.2 mm. was mounted on a glass fiber using epoxy cement such that the longest crystal dimension (b-axis) was approximately parallel to the

fiber axis.

Unit cell parameters and the orientation matrix were determined on a Syntex $P2_1$ four circle diffractometer equipped with a graphite monochromator (Bragg 2θ angle = 12.2°) using MoK_α radiation at a takeoff angle of 6.5°. Fifteen reflections whose 2θ values ranged from 4.00° to 50.00° were machine-centered and used in least-squares refinement of the lattice parameters and orientation matrix. Unit cell parameters obtained were a = 23.015(5)Å, b = 8.873(1)Å, c = 16.414(6)Å, α = 90°, β = 90°, γ = 90°, and V = 3351(2)Å^3. The calculated density of 1.62 g cm^{-3} for 8 formula units per unit cell agrees with the experimental density of 1.62 g cm^{-3} measured by the flotation method using a mixture of CCl_4 and 1,2-dibromo,1,1-dichloroethane. Omega scans of several low 2θ angle reflections gave peak widths at half-height of less than .24°, indicating a satisfactory mosaic spread for the crystal.

Axial photographs indicated that the crystal belonged to the orthorhombic system. Intensity data for zero and upper levels were collected at a rapid scan rate and the intensities examined carefully for systematic absences. The absence of k = 2n + 1 in 0kℓ, ℓ = 2n + 1 in hoℓ, and h+k = 2n + 1 in hk0 is consistent with only space group Pbcn (No. 60[b]).

Intensity data were collected using θ-2θ scans with x-ray source and monochromator settings identical to those used for determination of the unit cell parameters. A variable scan rate of from 3.5° to 29.3° min^{-1} was used and a scan width of 2.0° was sufficient to collect all of the peak intensity. Stationary background counts were measured at the beginning (bgd1) and at the end (bgd2) of each scan with a total background to scan time ratio, TR, of 1. No significant fluctuations were observed in the

intensities of three standard reflections (-5,5,0; -3,2,1; -8,0,-2) monitored every 100 reflections. Intensities were calculated from the total scan count (CT) and background counts by the relationship:

$$I = CT-(TR)(bdg1 + bgd2).$$

The intensities were assigned standard deviations according to the formula

$$\sigma(I) = [CT + (TR)^2(bgd1 + bgd2)]^{\frac{1}{2}}$$

from a total of 3498 reflections collected in a complete octant ($h,k,\ell=+$) of data out to $2\theta = 50.0°$; 2389 were accepted as statistically above background on the basis that I was greater than $2\sigma(I)$. Lorentz and polarization corrections were made in the usual way.

The positions of the copper atoms were deduced from a three-dimensional Patterson synthesis and refined; the resulting electron density map was used to locate the positions of eight other sets of non-hydrogen atoms which then refined to R=0.34. After the location of twenty-two non-hydrogen atoms, R was 0.17. Anisotropic refinement of these atoms lowered R to 0.08. Subsequent location of the disordered perchlorate oxygen atoms and eight hydrogen atoms gave R=0.046 and R_w=0.045.

The maximum shift in the final least squares cycle was 3.1 standard deviations on the β_{33} temperature factor of 08 of the disordered perchlorate group. The highest remaining electron density was located in the vicinity of the perchlorate group at about 25 percent of the value of refined positions.

Final positional and thermal parameters are listed in Table 6;

Table 6. Final Positional and Thermal Parameters for $Cu(L2pyr)(ClO_4)\cdot CH_3CN$

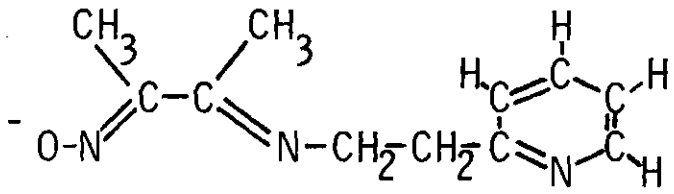
Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu	.07950(2)	.08487(5)	.23495(2)	.00128(1)	.01141(6)	.00266(2)	.00015(2)	.00007(1)	-.00032(3)
CL	.36664(5)	.01407(13)	.08959(7)	.00237(3)	.01682(18)	.00504(5)	.00135(6)	.00026(3)	-.00147(8)
O1	.0397 (1)	.1679 (3)	.3280 (1)	.0014 (1)	.0157 (4)	.0032 (1)	-.0003 (1)	.0002 (1)	-.0021 (2)
N1	.0148 (1)	.1282 (3)	.1558 (2)	.0015 (1)	.0092 (4)	.0029 (1)	-.0002 (1)	.0001 (1)	.0003 (2)
N2	.1218 (1)	.0573 (3)	.1306 (2)	.0016 (1)	.0103 (4)	.0032 (1)	.0001 (1)	.0003 (1)	-.0008 (2)
N3	.1572 (1)	.1121 (3)	.2974 (2)	.0015 (1)	.0104 (4)	.0035 (1)	-.0002 (1)	-.0002 (1)	.0006 (2)
C1	-.0048 (2)	.2173 (5)	.0163 (2)	.0020 (1)	.0182 (7)	.0032 (2)	.0000 (2)	-.0002 (1)	.0015 (3)
C2	.0326 (2)	.1483 (4)	.0820 (2)	.0017 (1)	.0089 (4)	.0027 (1)	-.0007 (1)	.0001 (1)	.0001 (2)
C3	.0939 (2)	.1008 (4)	.0683 (2)	.0020 (1)	.0081 (4)	.0031 (1)	-.0008 (1)	.0004 (1)	-.0006 (2)
C4	.1170 (2)	.1055 (7)	-.0174 (3)	.0022 (1)	.0198 (8)	.0039 (2)	-.0000 (2)	.0009 (1)	.0003 (3)
C5	.1827 (2)	.0059 (6)	.1237 (3)	.0017 (1)	.0155 (6)	.0044 (2)	.0006 (2)	.0006 (1)	.0001 (3)
C6	.2024 (2)	-.0670 (5)	.2031 (3)	.0016 (1)	.0128 (6)	.0051 (2)	.0010 (2)	.0003 (1)	.0003 (3)
C7	.2056 (1)	.0387 (4)	.2727 (3)	.0014 (1)	.0107 (5)	.0047 (2)	.0000 (1)	-.0001 (1)	.0016 (2)
C8	.2579 (2)	.0620 (5)	.3134 (3)	.0015 (1)	.0156 (7)	.0070 (2)	.0004 (2)	-.0008 (1)	.0012 (3)
C9	.2605 (2)	.1540 (6)	.3799 (3)	.0019 (1)	.0204 (8)	.0071 (3)	.0008 (2)	-.0014 (1)	.0010 (4)
C10	.2107 (2)	.2289 (5)	.4051 (3)	.0023 (1)	.0146 (6)	.0052 (2)	-.0017 (2)	-.0009 (1)	.0008 (3)
C11	.1602 (2)	.2050 (4)	.3608 (2)	.0018 (1)	.0112 (5)	.0040 (2)	-.0010 (2)	-.0003 (1)	.0005 (2)
NCN	.0687 (1)	-.1642 (4)	.2754 (2)	.0022 (1)	.0124 (5)	.0047 (2)	-.0004 (1)	-.0000 (1)	.0005 (2)
C12	.0642 (2)	-.2844 (5)	.2978 (2)	.0019 (1)	.0140 (6)	.0034 (1)	.0002 (2)	-.0002 (1)	-.0001 (3)
C13	.0576 (3)	-.4401 (5)	.3262 (3)	.0041 (1)	.0120 (7)	.0055 (2)	-.0001 (3)	-.0004 (1)	.0021 (3)
O2	.3280 (4)	.1250(10)	.1062 (6)	.005 (1)	.020 (1)	.010 (1)	.006 (1)	.002 (1)	.000 (1)
O3	.4132 (7)	-.0032(29)	.1310(10)	.005 (1)	.117 (7)	.011 (1)	.013 (1)	-.005 (1)	-.019 (2)
O4	.3814 (5)	-.0028(12)	.0072 (5)	.004 (1)	.037 (2)	.006 (1)	.000 (1)	.001 (1)	-.007 (1)
O5	.3212 (6)	-.1038(14)	.0839(11)	.005 (1)	.032 (2)	.020 (1)	-.002 (1)	.006 (1)	-.005 (1)
O6	.3660 (5)	-.0763(12)	.1629 (6)	.005 (1)	.028 (2)	.008 (1)	.003 (1)	.001 (1)	.004 (1)
O7	.3700(11)	.1687(11)	.0972 (9)	.015 (1)	.017 (2)	.014 (1)	-.001 (1)	.004 (1)	-.005 (1)

Table 6 (Continued). Final Positional and Thermal Parameters for $Cu(L2pyr)(ClO_4)\cdot CH_3CN$

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O8	.4197 (7)	-.0235(17)	.0637(17)	.005(1)	.036(3)	.030(2)	.004(1)	.009(1)	.008(2)
O9	.3313(14)	-.0452(39)	.0452(17)	.012(1)	.113(13)	.021(2)	.005(3)	.011(1)	-.008(5)
H5A	.207 (2)	.086 (5)	.106 (3)	5.0					
H5B	.191 (2)	-.067 (5)	.079 (3)	5.0					
H6A	.239 (2)	-.123 (5)	.204 (3)	5.0					
H6B	.180 (2)	.145 (5)	.221 (3)	5.0					
H8	.288 (2)	.006 (5)	.296 (3)	5.0					
H9	.295 (2)	.155 (5)	.413 (2)	5.0					
H10	.211 (2)	.287 (5)	.461 (3)	5.0					
H11	.126 (2)	.250 (5)	.378 (3)	5.0					
H4A	.109 (2)	.204 (6)	-.039 (3)	5.0					
H4B	.148 (2)	.089 (5)	-.023 (3)	5.0					

final calculated and observed structure factors are listed in the Appendix. Selected interatomic distances and angles are tabulated in Table 7, selected least squares planes are summarized in Table 8. The dimeric molecular unit is illustrated in Figure 5.

The eight formula units of $Cu(C2pyr)(ClO_4)\cdot CH_3CN$ in the unit cell form dimeric units about two-fold axes at (o,y,¼, (o,$\bar{y}$, 3/4), (½,½+y,¼), and (½,½-y,3/4), where y = .08487. Within the formula unit, the ligand



L2pyr

(L2pyr) is coordinated to the copper atom to form (1) a five-membered chelate ring involving the oxime nitrogen and the imine nitrogen, and (2) a six-membered chelate ring involving the imine nitrogen and the pyridine nitrogen. The fourth position in the coordination plane of the copper is occupied by the oxygen of the second ligand of the dimer, resulting in bridging oxime groups. The fifth coordination position of the square pyramid about copper is occupied by the weakly-bound acetonitrile nitrogen at 2.322(4) Å from the copper atom. The copper atom is 0.31 Å from the copper atom. The copper atom is 0.31 Å above the least squares plane of the coordinating atoms from (L2pyr) toward the axial ligand.

The most interesting feature of this structure is the oxime bridging arrangement. The six-membered ring is in a twisted boat conformation involving two coppers and two oxime (N-O) groups. The two basal

Table 7. Selected Interatomic Distances and Angles for $Cu(L2pyr)(ClO_4)\cdot CH_3CN$

Distances in Å			
Cu-Cu	3.693(2)	C12-N4	1.132(5)
Cu-O	1.928(2)	C1-C2	1.509(5)
Cn-N1	2.014(3)	C2-C3	1.489(5)
Cu-N2	1.986(3)	C3-C4	1.505(5)
Cu-N3	2.075(3)	C5-C6	1.525(6)
Cu-N4	2.322(4)	C6-C7	1.480(6)
N1-O'	1.329(4)	C7-C8	1.393(5)
C2-N1	1.292(4)	C8-C9	1.365(7)
C3-N2	1.268(5)	C9-C10	1.387(7)
C7-N3	1.352(5)	C10-C11	1.387(6)
C11-N3	1.330(5)	C12-C13	1.466(6)
Cℓ-O2	1.35 (1)	Cℓ-O6	1.45 (1)
Cℓ-O3	1.28 (1)	Cℓ-O7	1.38 (1)
Cℓ-O4	1.40 (1)	Cℓ-O8	1.33 (1)
Cℓ-O5	1.48 (1)	Cℓ-O9	1.21 (1)
C5-H5A	.96 (5)	C9-H9	.97 (4)
C5-H5B	1.00 (4)	C10-H10	1.06 (4)
C6-H6A	.98 (5)	C11-H11	.93 (5)
C6-H6B	.91 (5)	C4-H4A	.97 (5)
C8-H8	.90 (5)	C4-H4B	.73 (5)
Angles in Degrees			
O1-Cu-N1	95.0(1)	C3-N2-Cu	114.2(2)
N1-Cu-N2	80.2(1)	C3-N2-C5	120.9(3)
N2-Cu-N3	91.0(1)	C5-N2-Cu	124.7(2)
N3-Cu-O1	88.5(1)	N2-C5-C6	110.3(3)
N4-Cu-O1	94.9(1)	C5-C6-C7	114.6(4)
N4-Cu-N1	106.7(1)	C6-C7-C8	120.5(4)
N4-Cu-N2	100.5(1)	C6-C7-N3	119.7(3)
N4-Cu-N3	93.6(1)	N3-C7-C8	119.0(4)
N1'-O1-Cu	120.3(2)	C1-C8-C9	120.7(4)
Cu-N1-O1'	128.4(2)	C8-C9-C10	119.3(4)
Cu-N1-C2	113.3(2)	C9-C10-C11	117.5(4)
O1'-N1-C2	116.7(3)	C10-C11-N2	123.3(4)
N1-C2-C1	123.0(3)	C11-N3-Cu	120.3(3)
N1-C2-C3	113.8(3)	C11-N3-C7	119.3(3)
C1-C2-C3	123.2(3)	C7-N3-Cu	120.4(2)

Table 7 (Continued). Selected Interatomic Distances and Angles for $Cu(L2pyr)(ClO_4)\cdot CH_3CN$

Angles in Degrees			
C2-C3-C4	117.9(3)	C12-N4-Cu	177.6(3)
C2-C3-N2	116.4(3)	C13-C12-N4	179.2(4)
N2-C3-C4	125.6(4)		
O2-Cl-O3	122.0(9)	O6-Cl-O7	118.5(8)
O2-Cl-O4	115.6(7)	O6-Cl-O8	97.7(10)
O2-Cl-O5	93.6(7)	O6-Cl-O9	104.7(17)
O3-Cl-O4	107.2(9)	O7-Cl-O8	121.6(18)
O3-Cl-O5	122.7(15)	O7-Cl-O9	103.0(12)
O4-Cl-O5	92.0(7)	O8-Cl-O9	108.3(19)
H5A-C5-H5B	98 (3)	H8-C8-C9	123 (3)
H5A-C5-N2	111 (3)	H9-C9-C8	119 (3)
H5A-C5-N2	116 (3)	H9-C9-C10	121 (3)
H5B-C5-C6	113 (3)	H10-C10-C9	120 (2)
H5B-C5-C6	107 (3)	H10-C10-C11	122 (2)
H6A-C6-H6B	95 (3)	H11-C11-C10	119 (3)
H6A-C6-C5	106 (3)	H11-C11-N3	118 (3)
H6B-C6-C5	105 (3)	H4A-C4-H4B	109 (4)
H6A-C6-C7	118 (3)	H4A-C4-C3	108 (3)
H6B-C6-C7	115 (3)	H4B-C4-C3	117 (4)
H8-C8-C7	116 (3)		

Table 8. Least Squares Planes within the Molecule

$Cu(L2pyr)(ClO_4)\cdot CH_3CN$

Atom	Deviation Å	Atom	Deviation Å
(a) Plane involving four atoms strongly coordinated to copper:			
Equation: -0.17820x - 0.96690y + 0.18259z + 0.66089 = 0			
O1	0.041	N3	-0.054
N1	-0.033	Cu	0.311
N2	0.061		
(b) Plane involving the six atom imino oximo butane fragment of (L2pyr):			
Equation: 0.32505x + 0.92379y + 0.20237z - 1.74549 = 0			
N1	-0.065	C3	0.003
N2	0.069	C4	-0.060
C1	0.057	Cu	0.255
C2	-0.015		

[a]Direction cosines of the plane refer to the orthogonal axis system a,b,c*.

[b]All atoms weighted at unity.

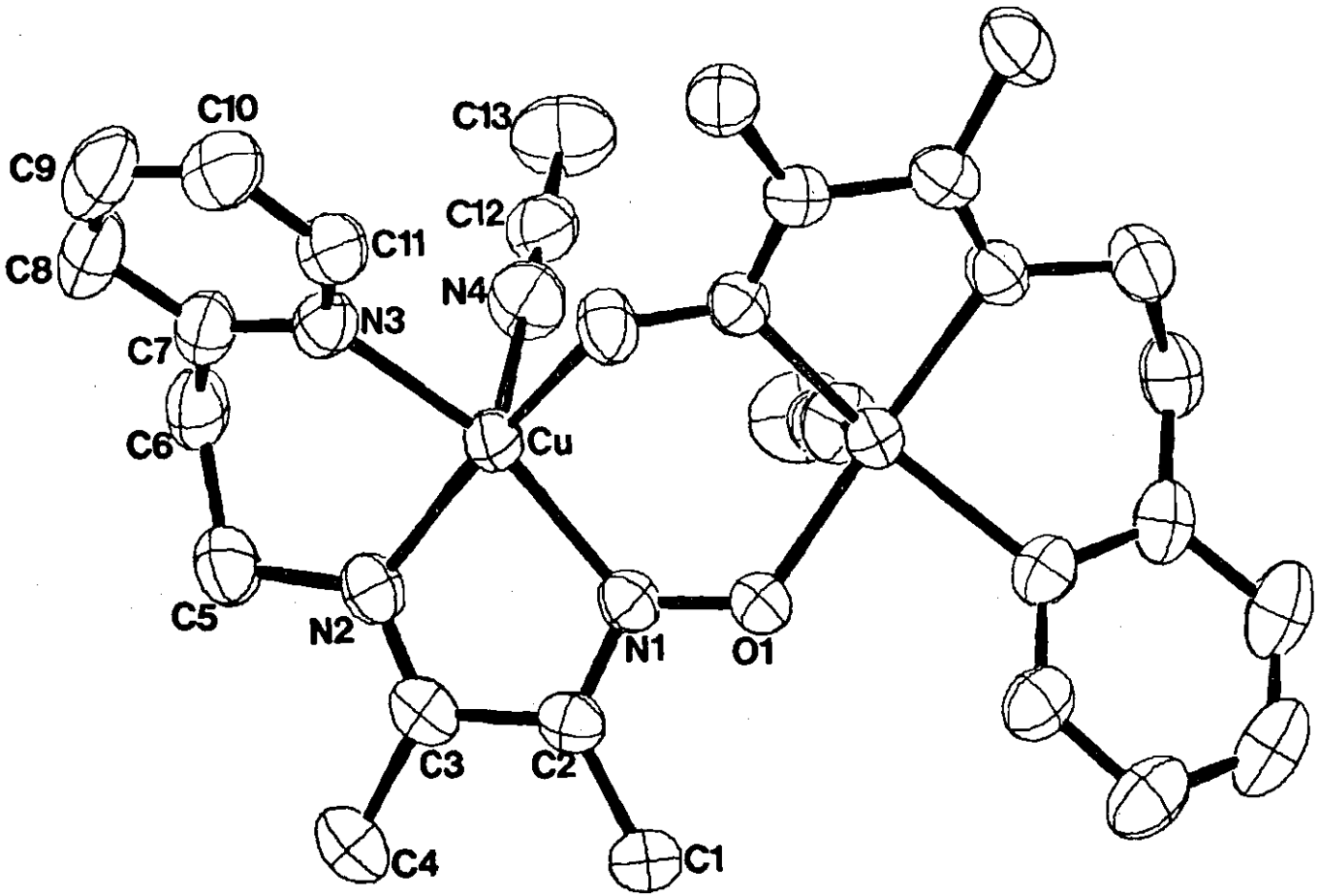


Figure 5. Dimeric Molecular Unit of $Cu(L2pyr)ClO_4 \cdot CH_3CN$

Omitting the Perchlorate Groups.

coordination planes are twisted 32 degrees to form the twisted boat conformation. The Cu-O1(1.928(2)Å) and Cu-N1(2.014(3)Å) distances are not as short as in the planar ring of $Cu(L2a\ell)(ClO_4)\cdot H_2O$, but nonetheless indicate strongly coordinated bridging oxime groups. The Cu-O1-N1' angle, where the primed quantities indicate atoms related by the two fold axis, is 120.3(2)°; and the Cu-N1-O1' angle is 128.4(2)°. These angles indicate essentially sp^2 hybridization for the oxime atoms.

The acetonitrile molecule, whose nitrogen is the fifth coordinating atom at a distance of 2.322(4)Å, shows fairly strong axial coordination; the N4-Cu-basal angles range from 93.6(1) to 106.7(1)°.

The six atom 2-imino-3-oximo butane fragment of (L2pyr) adopts a configuration which is almost planar, the maximum deviation from the least squares plane being 0.07 Å for N1. The C2-C3 distance of 1.489(5)Å and the C2-N1 and C3-N2 distances of 1.292(4) and 1.268(5)Å, respectively, agree well with the corresponding distances in dimethylglyoxime structures[7] and indicate the absence of cyclic delocalization. These segments are folded down away from the axial nitrile positions in the molecule.

The perchlorate ions are disordered and were refined with two sets of oxygen positions, each with multipliers of 0.5. No unusual inter- or intramolecular contacts other than those noted above, were observed.

<u>$Cu(L2am)(ClO_4)$</u>

A dark green crystal with approximate dimensions 0.2 x 0.3 x 0.7 mm. was mounted on a glass fiber using epoxy cement such that the longest crystal dimension (a axis) was approximately parallel to the fiber axis.

Unit cell parameters and the orientation matrix were determined on a Syntex $P2_1$ four circle diffractometer equipped with a graphite

monochromator (Bragg 2θ angle = 12.2°) using MoK_α radiation at a takeoff angle of 6.5°. Fifteen reflections whose 2θ values ranged from 3.51° to 21.89° were machine-centered and used in least-squares refinement of the lattice parameters and orientation matrix. Unit cell parameters obtained were a = 11.576(2)Å, b = 10.238(2)Å, c = 12.838(3)Å, α = 123.26(2)°, β = 98.84(1)°, γ = 126.31(1)°, and V = 1109.0(4)Å^3. The calculated density of 1.80 g cm^{-3} for 4 formula units per unit cell agrees with the experimental density of 1.82 g cm^{-3} measured by the flotation method using a mixture of carbon tetrachloride and 1,2-dibromo-1,1-dichloroethane. Omega scans of several low 2θ angle reflections gave peak widths at half-height of less than .27°, indicating a satisfactory mosaic spread for the crystal.

Axial photographs indicated that the crystal belonged to the triclinic system. Space group $P\bar{1}$ was assumed and the successful refinement of the structure has confirmed this choice.

Intensity data were collected using θ-2θ scans with x-ray source and monochromator settings identical to those used for determination of the unit cell parameters. A variable scan rate of from 29.30° to 7.32° min^{-1} was used and a scan width of 2.32° was sufficient to collect all of the peak intensity. Stationary background counts were measured at the beginning (bgd1) and at the end (bgd2) of each scan with a total background to scan time ratio, TR, of 1. No significant fluctuations were observed in the intensities of one standard reflection (-2,0,3) monitored every 100 reflections. Intensities were calculated from the total scan count (CT) and background counts by the relationship:

$$I = CT-(TR)(bdg1 + bgd2).$$

The intensities were assigned standard deviations according to the formula

$$\sigma(I) = [CT + (TR)^2(bgd1 + bgd2)]^{\frac{1}{2}}$$

from a total of 4140 reflections collected in a complete hemisphere ($\pm h, \pm k, + \ell$) of data out to $2\theta = 50°$; 3387 were accepted as statistically above background on the basis that I was greater than $2\sigma(I)$ Lorentz and polarization corrections were made in the usual way.

The 14 crystal faces were identified by optical means as the following (perpendicular distance in millimeters from the crystal center to the faces are given in parentheses): {010} (.185), {001} (.255), {$\bar{1}$11} (.379), {$\bar{1}$01} (.404), {$\bar{1}$1$\bar{1}$} (.535), {100} (.574), {$\bar{2}$10} (.663). Absorption corrections were calculated by the Guassian quadrature methods; corrections to F^2 ranged from 1.44 to 1.80.

Two independent copper atoms and two independent chlorine atoms were located in the three-dimensional Patterson synthesis and refined to R=0.34. Location of all 32 non-hydrogen atoms from electron density maps refined to R=0.12. Anisotropic refinement of all non-hydrogen atoms and isotropic refinement of all hydrogen atoms gave the final values of R=0.038 and $R_w = 0.040$.

The greatest shift in the final refinement was 3.2 standard deviations for one of the position parameters of one of the hydrogen atoms of a methyl group. Residual electron density was located largely in the vicinity of the perchlorate groups.

Final atomic positions and thermal parameters appear in Table 9, selected interatomic distances and angles are in Table 10, and least squares planes within the molecule are in Table 11. An illustration of

Table 9. Final Atomic Positional and Thermal Parameters for $Cu(L2am)(ClO_4)$

Unit A

Atom	x	y	z	β or β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu	.31316(5)	.02993(6)	.18046(3)	.01094(7)	.01209(8)	.00417(3)	.00736(7)	.00028(4)	.00158(4)
Cℓ	.2064 (2)	-.0934 (1)	.423 (1)	.0227 (2)	.0174 (2)	.0055 (1)	.0143 (2)	.0041 (1)	.0046 (1)
O1	.1626 (3)	.1620 (3)	.0915 (2)	.0104 (4)	.0142 (5)	.0043 (2)	.0081 (4)	.0014 (2)	.0025 (2)
N1	.2778 (3)	.1968 (4)	.1715 (2)	.0093 (4)	.0124 (5)	.0042 (2)	.0064 (4)	.0020 (2)	.0028 (3)
N2	.4938 (4)	.2454 (4)	.3105 (2)	.0116 (5)	.0150 (6)	.0043 (2)	.0076 (5)	.0001 (3)	.0019 (3)
N3	.4135 (5)	-.0683 (5)	.2203 (3)	.0168 (6)	.0197 (7)	.0059 (3)	.0133 (6)	.0022 (3)	.0034 (4)
C1	.3782 (6)	.5002 (6)	.2561 (4)	.0148 (7)	.0138 (8)	.0064 (3)	.0086 (6)	.0014 (4)	.0017 (4)
C2	.3800 (4)	.3554 (5)	.2509 (3)	.0103 (5)	.0125 (7)	.0041 (2)	.0068 (5)	.0022 (3)	.0024 (3)
C3	.5078 (4)	.3815 (5)	.3298 (3)	.0108 (5)	.0132 (7)	.0039 (2)	.0061 (5)	.0018 (3)	.0025 (3)
C4	.6431 (6)	.5507 (6)	.4207 (4)	.0138 (7)	.0147 (8)	.0075 (4)	.0067 (6)	-.0012 (4)	.0006 (4)
C5	.6036 (6)	.2319 (7)	.3687 (4)	.0147 (7)	.0211(10)	.0062 (3)	.0112 (8)	-.0008 (4)	.0024 (5)
C6	.5788 (6)	.0807 (7)	.2829 (4)	.0156 (7)	.0269(11)	.0076 (4)	.0155 (8)	.0014 (4)	.0053 (5)
O2	.1768 (5)	-.0643 (6)	.3223 (3)	.026 (1)	.045 (1)	.012 (1)	.027 (1)	.011 (1)	.019 (1)
O3	.2866 (5)	-.1585 (5)	.4117 (3)	.026 (1)	.030 (1)	.011 (1)	.021 (1)	.006 (1)	.010 (1)
O4	.0616 (5)	-.2258 (6)	.4298 (4)	.031 (1)	.033 (1)	.013 (1)	.021 (1)	.013 (1)	.013 (1)
O5	.2960 (8)	.0639 (6)	.5231 (4)	.051 (1)	.025 (1)	.013 (1)	.023 (1)	.004 (1)	-.001 (1)
H1C1	.3571 (6)	.523 (7)	.317 (4)	5.0					
H2C1	.462 (6)	.604 (7)	.286 (4)	5.0					
H3C1	.305 (6)	.458 (7)	.195 (5)	5.0					
H1C4	.679 (6)	.537 (7)	.486 (4)	5.0					
H2C4	.627 (6)	.629 (7)	.430 (4)	5.0					
H3C4	.728 (6)	.588 (7)	.412 (4)	5.0					
H1C5	.708 (6)	.342 (7)	.398 (4)	5.0					
H2C5	.581 (6)	.216 (7)	.434 (4)	5.0					
H1C6	.633 (6)	.043 (7)	.308 (4)	5.0					
H2C6	.614 (6)	.100 (7)	.225 (4)	5.0					
H1N3	.368 (6)	-.129 (7)	.260 (4)	5.0					
H2N3	.389 (6)	-.149 (7)	.163 (4)	5.0					

Table 9 (Continued). Final Atomic Positional and Thermal Parameters for Cu(L2am)(ClO_4)

Unit B

Atom	x	y	z	β or β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu	.12005(5)	.02822(5)	-.06384(3)	.01095(7)	.01131(8)	.00395(3)	.00715(6)	.00233(3)	.00296(4)
Cℓ	-.46168(12)	.30753(13)	.03160(9)	.0132 (2)	.0136 (2)	.0087 (1)	.0075 (1)	.0054 (1)	.0048 (1)
O1	.1338 (3)	-.1842 (3)	.0572 (2)	.0138 (4)	.0123 (5)	.0040 (2)	.0073 (4)	.0003 (2)	.0025 (2)
N1	.1100 (3)	-.1649 (4)	-.0422 (2)	.0094 (4)	.0106 (5)	.0041 (2)	.0057 (4)	.0012 (2)	.0026 (3)
N2	.0583 (4)	-.1162 (4)	-.2225 (2)	.0122 (5)	.0143 (6)	.0043 (2)	.0086 (5)	.0030 (3)	.0037 (3)
N3	.1281 (4)	.1925 (4)	-.1305 (3)	.0135 (5)	.0126 (6)	.0061 (2)	.0077 (5)	.0035 (3)	.0045 (3)
C1	.0199 (7)	-.4612 (6)	-.1401 (4)	.0232 (9)	.0132 (8)	.0061 (3)	.0117 (7)	.0001 (4)	.0019 (4)
C2	.0568 (4)	-.2943 (5)	-.1364 (3)	.0087 (5)	.0110 (6)	.0046 (2)	.0053 (5)	.0015 (3)	.0025 (3)
C3	.0339 (4)	-.2595 (5)	-.2407 (3)	.0086 (5)	.0120 (6)	.0045 (2)	.0057 (5)	.0017 (3)	.0022 (3)
C4	-.0163 (6)	-.3909 (6)	-.3559 (3)	.0206 (8)	.0160 (8)	.0045 (3)	.0111 (7)	.0030 (4)	.0023 (4)
C5	.0405 (6)	-.0481 (6)	-.3068 (3)	.0185 (7)	.0203 (9)	.0047 (3)	.0136 (7)	.0045 (4)	.0058 (4)
C6	.1457 (6)	.1505 (6)	-.2424 (4)	.0159 (7)	.0176 (8)	.0065 (3)	.0099 (7)	.0045 (4)	.0065 (4)
O2	-.3531 (4)	.3669 (5)	-.0317 (3)	.020 (1)	.028 (1)	.010 (1)	.015 (1)	.009 (1)	.007 (1)
O3	-.3955 (9)	.4430 (6)	.1275 (6)	.064 (2)	.019 (1)	.026 (1)	.005 (1)	.034 (1)	.002 (1)
O4	-.4857(12)	.1691(11)	.0535 (7)	.094 (3)	.063 (2)	.033 (1)	.066 (3)	.040 (2)	.036 (1)
O5	-.5915 (7)	.2442(16)	-.0326 (8)	.019 (1)	.126 (5)	.039 (1)	.031 (2)	.015 (1)	.048 (2)
H1C1	.058 (6)	-.511 (7)	-.201 (4)	5.0					
H2C1	-.072 (6)	-.551 (7)	-.173 (4)	5.0					
H3C1	.077 (6)	-.438 (7)	-.076 (4)	5.0					
H1C4	-.155 (6)	-.513 (7)	-.373 (4)	5.0					
H2C4	-.022 (6)	-.368 (7)	-.409 (4)	5.0					
H3C4	.042 (6)	-.434 (7)	-.356 (4)	5.0					
H1C5	.072 (5)	-.078 (7)	-.366 (4)	5.0					
H2C5	-.072 (6)	-.103 (7)	-.336 (4)	5.0					
H1C6	.115 (6)	.203 (7)	-.282 (4)	5.0					
H2C6	.248 (6)	.200 (7)	-.224 (4)	5.0					
H1N3	.046 (6)	.185 (7)	-.138 (4)	5.0					
H2N3	.214 (6)	.314 (7)	-.077 (4)	5.0					

Table 10. Selected Interatomic Distances and Angles in $Cu(L2am)(ClO_4)$

Unit A	Distances in Å	Unit B
1.905(3)	Cu-O1'	1.935(3)
2.001(3)	Cu-N1	1.997(3)
1.950(3)	Cu-N2	1.940(3)
2.015(3)	Cu-N3	2.017(3)
1.345(4)	O1-N1	1.347(4)
1.292(5)	C2-N1	1.291(5)
1.261(5)	C3-N2	1.271(5)
1.470(5)	C5-N2	1.467(5)
1.463(6)	C6-N3	1.484(5)
1.481(6)	C1-C2	1.482(6)
1.494(5)	C2-C3	1.487(5)
1.495(6)	C3-C4	1.494(5)
1.512(7)	C5-C6	1.513(6)
1.422(3)	Cℓ-O2	1.430(3)
1.328(5)	Cℓ-O3	1.434(4)
1.341(6)	Cℓ-O4	1.435(4)
1.350(6)	Cℓ-O5	1.407(5)
.88 (5)	C1-HC1A	1.12 (5)
.84(5)	C1-HC1B	.82 (5)
.85(5)	C1-HC1C	.85 (5)
1.02(5)	C4-HC4A	1.00 (5)
.82(5)	C4-HCHB	1.01 (5)
.88(5)	C4-HCHC	.78 (5)
.96(5)	C5-HC5A	.96 (5)
.93(5)	C5-HC5B	1.01 (5)
.96(5)	C6-HC6A	1.00 (5)
.91(5)	C6-HC6B	.93 (5)
.86(5)	N3-HN3A	.90 (5)
.82(5)	N3-HN3B	.97 (5)

CuA-CuB	3.550(2)
CuB-CuBI	3.268(2)
CuA-O2A	2.574(4)
CuB-O1'I	2.530(4)

'indicates in the adjacent unit of the asymmetric dimer.

Irelated by the inversion center of the tetramer.

Table 10 (Continued). Selected Interatomic Distances and Angles in $Cu(L2am)(ClO_4)$

Unit A	Angles in Degrees	Unit B
79.9(1)	N1-Cu-N2	80.1(1)
83.3(2)	N2-Cu-N3	84.0(1)
96.1(2)	N3-Cu-O1'	95.6(1)
100.9(1)	N1-Cu-O1'	100.6(1)
127.1(2)	Cu-N1-O1	127.2(2)
112.9(2)	Cu-O1'-N1'	113.4(2)
117.8(3)	O1-N1-C2	117.4(3)
112.9(3)	N1-C2-C1	112.9(3)
123.3(3)	N1-C2-C3	123.5(3)
123.6(3)	C1-C2-C3	123.6(3)
120.3(4)	C2-C3-C4	119.5(3)
115.2(3)	C2-C3-N2	115.2(3)
124.4(4)	C4-C3-N2	125.3(3)
127.8(3)	C3-N2-C5	128.3(3)
106.9(3)	N2-C5-C6	106.2(3)
109.9(4)	C5-C6-N3	109.7(3)
115.1(2)	C2-N1-Cu	114.8(2)
116.8(3)	C3-N2-Cu	116.5(2)
115.2(3)	C5-N2-Cu	115.2(3)
107.8(3)	C6-N3-Cu	106.7(3)
	N1'-O1'CuI	88.8(1)
	O1'-Cu-O1'I	86.9(1)
	N1-Cu-O1'I	87.0(1)
	N2-Cu-O1'I	88.2(1)
	N3-Cu-O1'I	96.5(1)
118.8(1)	O1'-Cu-O2	
95.6(1)	N1-Cu-O2	
88.5(2)	N2-Cu-O2	
88.6(1)	N3-Cu-O2	
108.1(2)	O2-Cℓ-O3	109.6(3)
108.7(2)	O2-Cℓ-O4	116.2(4)
111.3(3)	O2-Cℓ-O5	108.7(4)
108.8(3)	O3-Cℓ-O4	108.0(7)
109.8(3)	O3-Cℓ-O5	111.4(5)
110.1(3)	O4-Cℓ-O5	102.5(6)

'Indicates atoms in the adjacent unit of the asymmetric dimer.

IIndicates atoms related by the center of inversion in the tetramer.

Table 10 (Concluded). Selected Interatomic Distances and Angles in $Cu(L2am)(ClO_4)$

Angles in Degrees		
Unit A		Unit B
88(4)	H1C1-C1-H2C1	93(4)
111(5)	H1C1-C1-H3C1	100(4)
124(5)	H1C1-C1-C2	114(3)
106(3)	H2C1-C1-H3C1	124(4)
119(3)	H2C1-C1-C2	115(4)
106(3)	H3C1-C1-C2	108(3)
99(4)	H1N3-N3-H2N3	104(4)
114(3)	H1N3-N3-C6	112(3)
117(3)	H2N3-N3-C6	112(3)
111(3)	H2N3-N3-Cu	114(3)
108(3)	H1N3-N3-Cu	109(3)
123(4)	H1C4-C4-H2C4	109(4)
81(4)	H1C4-C4-H3Cr	91(4)
122(5)	H1C4-C4-C3	112(3)
105(3)	H2C4-C4-H3C4	110(4)
87(3)	H2C4-C4-C3	118(4)
108(3)	H3C4-C4-C3	113(3)
105(4)	H1C5-C5-H2C5	113(4)
111(3)	H1C5-C5-N2	108(3)
113(3)	H1C5-C5-C6	111(3)
104(3)	H2C5-C5-N2	107(3)
115(3)	H2C5-C5-C6	111(3)
94(4)	H1C6-C6-H2C6	116(4)
112(3)	H1C6-C6-N3	106(3)
119(3)	H1C6-C6-C5	112(3)
103(3)	H2C6-C6-N3	103(3)
117(3)	H2C6-C6-C5	110(3)

' Indicates atoms in the adjacent unit of the asymmetric dimer.

I Indicates atoms related by the center of inversion in the tetramer.

Table 11. Least Squares Planes within the Molecule

$[Cu(L2am)(ClO_4)_4]^{a,b}$

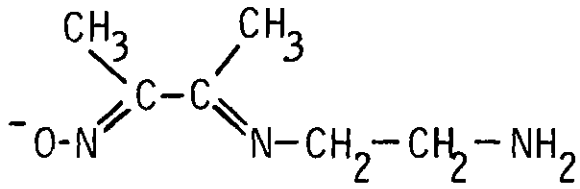
Atom	Deviation Å	Atom	Deviation Å
(a) Plane involving four atoms strongly coordinated to CuA. Equation: -0.64055x - 0.49684y + 0.58553z + 0.71778 = 0			
O1B	0.312	C5A	-0.291
N1A	0.252	C6A	-0.893
N2A	-0.033	CuA	0.110
N3A	-0.231		
(b) Plane involving four atoms strongly coordinated to CuB. Equation: 0.86337x + 0.50331y + 0.03576z - 1.30712 = 0			
O1A	-0.071	C5B	-0.136
N1B	0.068	C6B	0.551
N2B	-0.082	CuB	0.011
N3B	0.068		
(c) Plane involving six atom imino-oximo-butane fragment of (L2am)A Equation: -0.55848x - 0.49978y + 0.66206z + 0.17078 = 0			
C1A	0.003	N1A	0.002
C2A	0.045	N2A	0.006
C3A	0.015	CuA	-0.011
C4A	-0.046		
(d) Plane involving six atom imino oximo butane fragment of (L2am)B Equation: 0.89649x + 0.44296y + 0.00911z - 1.42991 = 0			
C1B	-0.039	N1B	0.011
C2B	0.037	N2B	-0.041
C3B	0.008	CuB	-0.069
C4B	0.020		

[a]Direction cosines of the plane refer to the orthogonal axis system a,b,c*.

[b]All atoms weighted at unity.

the tetrameric unit appears in Figure 6. Final calculated and observed structure factors appear in the Appendix.

The four formula units of $Cu(L2am)(ClO_4)$ in the unit cell form a centrosymmetric tetramer about the origin. The tetrameric unit, which can be thought of as two dimers joined together, is illustrated in Figure 6. Within the asymmetric dimer (unit A and unit B in Figure 6) the two copper atoms have similar coordination geometries. A ligand L2am



L2am

is coordinated to each copper to form (1) a five-membered chelate ring involving the oxime nitrogen and the imine nitrogen and (2) a five-membered chelate ring involving the imine nitrogen and the amine nitrogen. The fourth position in the basal portion of the square pyramidal coordination is the oxygen of the ligand bound to the other copper atom of the dimer, resulting in bridging oxime groups; the six-membered ring formed by the bridging oxime groups is a twisted boat conformation. The fifth coordination site of each copper atom is occupied by an oxygen atom but the nature of these oxygens is different. In one case (CuA) the axial atom is an oxygen atom of a perchlorate group and in the other case (CuB) the axial atom is an oxime oxygen from the symmetry related dimer, forming a planar (symmetry required) four-membered Cu_2O_2 ring. The coordination of CuB is square pyramidal with the copper atom 0.01 Å above the least

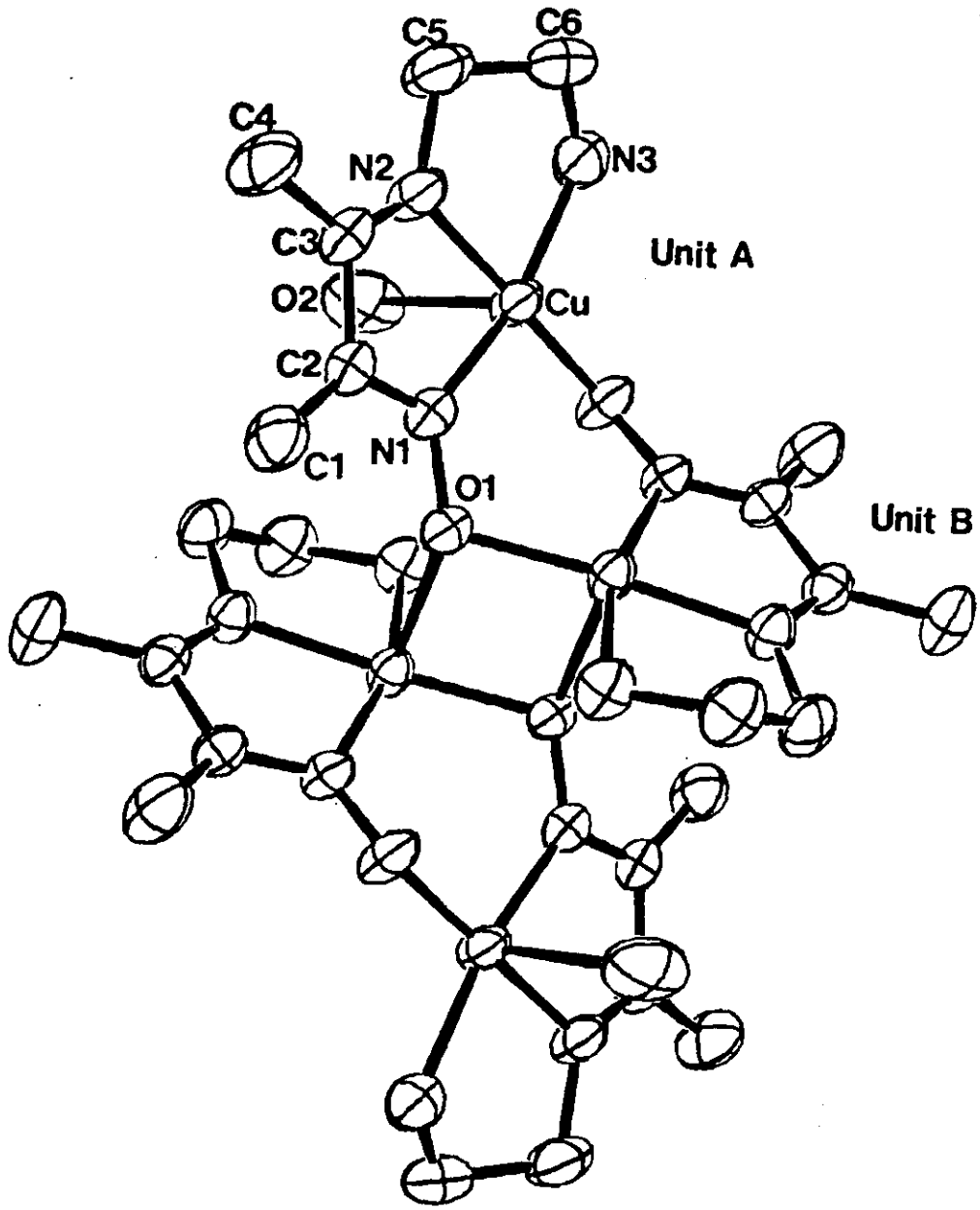


Figure 6. Tetrameric Molecular Unit of $Cu(L2am)(ClO_4)$ Omitting the Perchlorate Groups.

squares plane formed by the four basal coordinating atoms (maximum deviation of these from the best plane was -0.08 Å for the imine nitrogen). For CuA, the four strongly coordinated atoms are not so rigidly planar and the copper atom is 0.11 Å out of the basal plane toward the axial oxygen (maximum deviation from the least squares plane involving the four basal coordinating atoms of CuA is 0.31 Å for the oxime oxygen O1B). This copper appears to be distorted slightly toward a trigonal bipyramidal configuration. The axial copper-oxygen distances of 2.530(4) Å for the bridging oxygen and 2.574(4) Å for the perchlorate oxygen indicate weak interactions in the same range as the copper-oxygen (water) axial interaction in $Cu(L2A\ell)(ClO_4)\cdot H_2O$. The bridging oxygen-copper-basal angles range from 86.9(1) to 96.5(1)° and the perchlorate oxygen-copper-basal angles range from 88.5(2) to 111.8(1)°.

The most interesting feature of this structure is the six-membered ring resulting from the oxime bridging arrangement. Unlike $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, there is no symmetry imposed on the ring; however, a comparison of the two ring systems, and the basal coordination planes of the copper atoms in each dimer, reveals a great structural similarity. The two basal planes are twisted to form a twisted boat conformation as in $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, but only the angles at the nitrogen atom are comparable for the two complexes, Table 12. The CuA-O1B (1.905(3)Å), CuB-O1A (1.935(3)Å), CuA-N1A (2.001(3)Å), and CuB-N1B (1.997(3)Å) are not as short as in the planar ring of $Cu(L2a\ell)(ClO_4)(H_2O)$ but, like $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, they indicate a strongly coordinated bridging group. The CuA-O1B-N1B (112.9(2)°) and CuB-O1A-N1A (113.4(2)°) angles indicate a predicted variation toward sp^3 hybridization from coordination

Table 12. Oxime Bridging Ring Angles and Geometry, Copper-Copper Distances, Copper Coordination and Room and Low Temperature Magnetic Moments of Bridging Oxime Complexes

Complex	Ring Angles			Copper-Copper Distance	Copper Coordination	Ring Geometry	Magnetic Moment	
	N-Cu-O	CuO-N	O-N-Cu				298°	93°
$Cu(dmgH)_2$	91.6(1)	102.5(2)	122.8(3)	3.851	sq. pyr	chair	1.79	1.76
Cu(4,4,9,9)Br	92.2(4)	122.2(7)	114.0(8)	3.949(4)	tbp.	boat	1.70	
$Cu(L2al)(ClO_4(H_2O)$	108.3(2)	121.9(3)	129.4(3)	3.581(3)	oct.*	planar	0.26	0.20
$Cu(L2pyr)(ClO_4)\ CH_3CN)$	95.1(1)	120.1(2)	128.3(2)	3.693(2)	sq. pyr.	tw. boat	0.65	0
$Cu(L2am)(ClO_4)$ unit A	100.9(1)	112.9(2)	127.1(2)	3.550(2)	sq. pyr.	tw. boat	1.36	0.34
unit B	100.6(1)	113.4(2)	127.2(2)		sq. pyr.			
$Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$	-	141.7(3)	-	5.129(4)	oct.*	-	1.77	1.71

*Tetragonally distorted.

to the second copper atom. The CuA-N1A-O1A (127.1(2)°), and CuB-N1B-O1B (127.2(2)°) angles indicate essentially sp^2 hybridization.

The six atom 2-imino-3-oximo-butane fragment of (L2am) on each copper atom adopts a nearly planar configuration (the maximum deviation from the best plane in Unit A of the asymmetric dimer is 0.04 Å for C2, and in Unit B, 0.04 Å for N2). The C2-C3 distances of 1.494(5) and 1.487(5) Å, the C2-N1 distances of 1.292(5) and 1.291(5) Å, and the C3-N2 distances of 1.261(5) and 1.271(5) Å, for Unit A and Unit B respectively, agree well with the corresponding distances in dimethylglyoxime structures[7] and indicate the absence of cyclic delocalization.

The ethylenediamine portion of the ligand (L2am) adopts a twist conformation similar to that found in many ethylenediamine chelate rings. In Unit A, C5 and C6 lie 0.14 Å above and 0.55 Å below, respectively, the least squares plane of the four strongly coordinating atoms and in Unit B, 5 and 6 lie 0.29 Å and 0.89 Å, respectively, below the least squares plane of the four coordinating atoms.

The perchlorate anion which is not weakly coordinated to CuA appears to be associated with N3A by hydrogen bonding to O2B. The O2B-N3A distance is 3.041(6) Å and H1N3B - O2B is 2.228(5) Å. Further evidence is the comparison of the lengths of the principle axes of the thermal ellipsoids. The average of the first and second principal axes of O3B, O4B, and O5B, are 18% and 17% higher than the same axes of O2B, however, the average length of the third principal axis of O3B, O4B, and O5B, was 2.24 times the length of the third principal axis of O2B. ORTEP plots show that the O3B, O4B, and O5B atoms appear to be vibrating around the CLB-O2B axis.

$\underline{Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH}$

A wine red crystal with approximate dimensions 0.3 x 0.3 x 0.6 mm. was mounted on a glass fiber using epoxy cement such that the longest crystal dimension (z axis) was approximately parallel to the fiber axis.

Unit cell parameters and the orientation matrix were determined on a Syntex $P2_1$ four circle diffractometer equipped with a graphite monochromator (Bragg 2θ angle = 12.2°) using MoK_α radiation at a takeoff angle of 6.5°. Fifteen reflections whose 2θ values ranged from 25.3° to 10.7° were machine-centered and used in least-squares refinement of the lattice parameters and orientation matrix. Unit cell parameters obtained were a = 6.897(3)Å, b - 12.023(4)Å, c = 22.646(7)Å, α = 100.81(3)°, β = 98.69(3)°, γ = 106.73(3)°, and V = 1724(1)Å^3. The calculated density of 1.61 g cm^{-3} for 4 formula units per unit cell agrees with the experimental density of 1.60 g cm^{-3} measured by the flotation method using a mixture of carbon tetrachloride and 1,2 dibromo-1,1 dichloroethane. Omega scans of several low 2θ angle reflections gave peak widths at half-height of less than .25°, indicating a satisfactory mosaic spread for the crystal.

Axial photographs indicated that the crystal belonged to the triclinic system. Space group $P\bar{1}$ was assumed and the successful refinement of the structure has confirmed this choice.

Intensity data were collected using θ-2θ scans with x-ray source and monochromator settings identical to those used for determination of the unit cell parameters. A variable scan rate of from 5.0° to 29.3° min was used and a scan width of 2° was sufficient to collect all of the peak intensity. Stationary background counts were measured at the beginning

(bgd1) and at the end (bgd2) of each scan with a total background to scan time ratio, TR, of 1. No significant fluctuations were observed in the intensities of three standard reflections (0,3,-5; 1,4,-1;2,2,-4) monitored every 100 reflections. Intensities were calculated from the total scan count (CT) and background counts by the relationship:

$$I = CT-(TR)(bdg1 + bgd2).$$

The intensities were assigned standard deviations according to the formula

$$\alpha(I) = [CT + (TR)^2(bgd1 + bgd2) + (pI)^2]^{\frac{1}{2}}$$

from a total of 6291 reflections collected in a complete hemisphere ($\pm h, \pm k, + \ell$) of data out to $2\theta = 50°$; 4383 were accepted as statistically above background on the basis that I was greater than $3\sigma(I)$ with $p = 0$. Lorentz and polarization corrections were made in the usual way.

The ten crystal faces were identified by optical means as the following (perpendicular distances in millimeters from the crystal center to the faces are given in parentheses): $\{\bar{1}\bar{1}0\}$ (.167), $\{011\}$ (.164), $\{0\bar{1}1\}$ (.181), $\{1\bar{2}\bar{1}\}$ (.127), $\{001\}$ (.322). Absorption corrections were calculated by the Gaussian quadrature methods; corrections to F^2 ranged from 1.20 to 1.40.

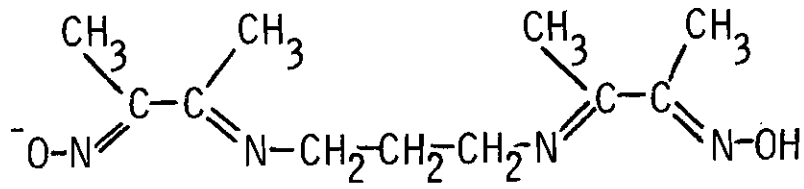
Two sets of copper atoms were located in a three-dimensional Patterson synthesis and a structure-factor calculation refined to R=.46. When all non-hydrogen atoms except solvent molecules and perchlorate oxygens were included, R was equal to 0.19. Introduction of the perchlorate oxygens and refinement of anisotropic thermal parameters for twelve atoms lowered R to 0.11. Final refinement, including two solvent molecules

with multipliers of 0.5, twelve hydrogen atoms in fixed positions and anisotropic thermal parameters for all non-hydrogen atoms gave R=0.060 and R_w=0.068.

Residual electron density was observed in the vicinity of solvent molecules and C6. The largest shift in the final refinement was 2.7 standard deviations for the z coordinate of the methanol carbon in unit B.

Final atomic positional and thermal parameters are listed in Table 13, selected interatomic distances and angles are in Table 14, and least squares planes of the two molecular units of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$ are in Table 15. An illustration of the monomeric unit appears in Figure 7 and a stereoview of the polynuclear chains appears in Figure 8. Calculated and observed structure factors appear in the Appendix.

The four formula units of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$ in the unit cell form two independent chains which are related by an inversion center at the origin; these chains are illustrated in the stereoview (Figure 9). Within each formula unit, the ligand (L3LH)



L3LH

is coordinated to copper to form (1) two five-membered chelate rings involving one oxime nitrogen and one imine nitrogen and (2) one six-membered chelate ring involving two imine nitrogens. The four nitrogen atoms of each ligand occupy the basal coordination positions (maximum deviation from the least squares planes is 0.03 Å for N4B) of a distorted

Table 13. Final Positional and Thermal Parameters for $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$

Unit A

Atom	x	y	z	β or β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu	.3876(1)	-.4115(1)	.2558(1)	.0275(2)	.0098(1)	.0016(1)	.0094(1)	.0021(1)	.0014(1)
Cℓ	.1634(3)	-.2596(2)	-.0692(1)	.0392(6)	.0100(2)	.0028(1)	.0060(3)	.0031(1)	.0009(1)
O1	.0904(6)	-.2890(4)	.2280(2)	.0210(11)	.0086(4)	.0020(1)	.0099(6)	.0023(3)	.0011(1)
N1	.2108(7)	-.3490(4)	.2053(2)	.0250(12)	.0070(4)	.0014(1)	.0050(6)	.0014(3)	.0008(2)
N2	.4476(7)	-.4685(4)	.1765(2)	.0231(12)	.0077(5)	.0019(1)	.0059(6)	.0021(3)	.0004(2)
N3	.5376(7)	-.4876(4)	.3073(2)	.0226(13)	.0075(5)	.0028(1)	.0063(6)	.0016(3)	.0018(2)
N4	.2896(7)	-.3754(4)	.3305(2)	.0229(12)	.0072(4)	.0017(1)	.0050(6)	.0016(3)	.0010(2)
C1	.0727(12)	-.3407(7)	.1101(2)	.0458(24)	.0131(8)	.0018(1)	.0119(11)	.0009(5)	.0015(3)
C2	.2060(8)	-.3734(5)	.1474(2)	.0224(15)	.0062(5)	.0018(1)	.0026(7)	.0010(3)	.0008(2)
C3	.3511(9)	-.4425(5)	.1319(3)	.0240(15)	.0064(5)	.0018(1)	.0022(7)	.0023(4)	.0001(2)
C4	.3698(13)	-.4719(8)	.0663(3)	.0445(24)	.0157(9)	.0016(1)	.0116(12)	.0030(5)	.0006(3)
C5	.5925(10)	-.5349(7)	.1707(3)	.0299(19)	.0121(8)	.0030(2)	.0096(10)	.0035(5)	.0008(3)
C6	.5859(15)	-.6125(8)	.2148(5)	.0511(30)	.0134(10)	.0045(3)	.0158(14)	.0054(7)	.0019(4)
C7	.6712(11)	-.5515(7)	.2829(3)	.0480(26)	.0153(9)	.0020(2)	.0109(12)	.0035(5)	.0025(3)
C8	.5859(14)	-.5351(9)	.4087(4)	.0377(20)	.0116(7)	.0031(2)	.0114(10)	.0027(5)	.0020(3)
C9	.5017(9)	-.4804(5)	.3613(3)	.0498(28)	.0177(11)	.0031(2)	.0160(14)	.0025(6)	.0042(4)
C10	.3582(9)	-.4121(5)	.3763(2)	.0238(15)	.0075(6)	.0021(1)	.0033(7)	.0007(4)	.0016(2)
C11	.3016(13)	-.3908(8)	.4372(3)	.0279(16)	.0067(5)	.0016(1)	.0031(7)	.0010(3)	.0011(2)
O3	.270 (2)	-.189 (1)	-.101 (1)	.101 (1)	.023 (1)	.007 (1)	.000 (2)	.014 (1)	.005 (1)
O4	.035 (2)	-.350 (1)	-.113 (1)	.100 (5)	.030 (2)	.010 (1)	-.017 (3)	.009 (2)	-.001 (1)
O5	.084 (3)	-.208 (1)	-.027 (1)	.222 (11)	.034 (2)	.009 (1)	.066 (4)	.033 (2)	.009 (1)
O6	.247 (3)	-.334 (2)	-.046 (1)	.245 (13)	.055 (3)	.007 (1)	.099 (6)	.020 (2)	.011 (1)
O(Sol)	.089 (2)	-.618 (1)	.228 (1)	.035 (3)	.015 (1)	.003 (1)	-.000 (1)	.000 (1)	.002 (1)
C(Sol)	.063 (2)	.289 (2)	.256 (1)	.034 (5)	.013 (2)	.005 (1)	.005 (2)	.001 (1)	.004 (1)
H5A	.61	-.58	.13	5.0					
H5B	.73	-.48	.18	5.0					
H6A	.67	-.68	.20	5.0					
H6B	.42	-.70	.21	5.0					
H7A	.82	-.48	.29	5.0					
H7B	.70	-.60	.31	5.0					

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Table 14. Selected Interatomic Distances and Angles of

$Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$

Unit A	Distances in Å	Unit B
1.947(5)	Cu-N1	1.944(5)
1.947(5)	Cu-N2	1.949(5)
1.961(5)	Cu-N3	1.960(5)
1.937(5)	Cu-N4	1.940(5)
2.493(4)	Cu-O1'	2.491(4)
2.624(10)	Cu-O(Sol)	2.625(11)
1.344(6)	N1-O1	1.345(6)
1.369(6)	N4-O2	1.373(6)
1.282(7)	C2-N1	1.282(7)
1.280(7)	C10-N4	1.267(7)
1.263(7)	C3-N2	1.259(7)
1.277(8)	C4-N3	1.278(8)
1.454(8)	C5-N2	1.465(8)
1.465(8)	C7-N3	1.461(8)
1.487(8)	C1-C2	1.487(8)
1.485(9)	C10-C11	1.491(9)
1.513(8)	C2-C3	1.510(8)
1.496(9)	C9-C10	1.500(9)
1.497(9)	C3-C4	1.493(8)
1.490(9)	C8-C9	1.487(9)
1.368(20)	C(Sol)-O(Sol)	1.412(20)
	CuA-CuB 5.129(4)	
1.349(8)	Cl-O3	1.342(8)
1.311(13)	Cl-O4	1.326(13)
1.323(9)	Cl-O5	1.313(9)
1.349(10)	Cl-O6	1.340(9)
1.020(7)	C5-H5a	0.986(7)
0.952(7)	C5-H5b	0.817(7)
1.156(8)	C6-H6a	1.213(11)
1.287(10)	C6-H6b	1.204(8)
1.099(8)	C7-H7a	1.099(8)
.958(7)	C7-H7b	.964(7)

Table 14 (Continued). Selected Interatomic Distances and Angles of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$

Unit A	Angles in Degrees	Unit B
81.6(2)	N1 Cu N2	81.4(2)
100.2(2)	N2 Cu N3	100.5(2)
80.6(2)	N3 Cu N4	80.4(2)
96.7(2)	N4 Cu N1	96.8(2)
84.4(2)	O1'Cu N1	84.5(2)
91.8(2)	O1'Cu N2	91.9(2)
101.5(2)	O1'Cu N3	101.5(2)
96.4(2)	O1'Cu N4	96.6(2)
120.6(4)	O1 N1 C2	120.8(4)
123.2(3)	O1 N1 Cu	123.1(3)
116.1(4)	Cu N1 C2	116.0(4)
125.0(5)	N1 C2 C1	124.7(5)
111.8(5)	N1 C2 C3	112.2(5)
123.2(5)	C1 C2 C3	123.1(5)
117.8(5)	C2 C3 C4	118.1(5)
116.1(5)	C2 C3 N2	115.6(5)
126.2(5)	C4 C3 N2	126.4(5)
114.3(4)	C3 N2 Cu	114.8(4)
124.0(5)	C3 N2 C5	123.8(5)
121.6(4)	Cu N2 C5	121.4(4)
111.7(6)	N2 C5 C6	111.9(5)
117.8(7)	C5 C6 C7	118.3(8)
111.1(6)	C6 C7 N3	110.8(6)
95.0(4)	C7 N3 C8	95.0(4)
121.1(4)	C7 N3 Cu	121.2(4)
114.7(4)	Cu N3 C9	114.6(4)
126.0(6)	N3 C9 C8	126.2(6)
115.3(5)	N3 C9 C10	115.2(5)
118.6(6)	C8 C9 C10	118.6(5)
123.6(5)	C9 C10 C11	123.2(5)
111.9(5)	C9 C10 N4	112.0(5)
124.5(6)	C11 C10 N4	124.8(5)
117.4(4)	C10 N4 Cu	117.7(4)
119.4(4)	C10 N4 O2	119.6(4)
123.1(3)	Cu N4 O2	122.6(3)
141.7(3)	Cu'O1 N1	141.7(3)
89.7(3)	O(Sol) Cu N1	89.3(3)
85.1(3)	O(Sol) Cu N2	85.0(3)
84.5(3)	O(Sol) Cu N3	84.8(3)
86.4(3)	O(Sol) Cu N3	86.3(3)

Table 14 (Concluded). Selected Interatomic Distances and Angles of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$

Angles in Degrees		
Unit A		Unit B
102.4(9)	O3 Cl O4	102.1(9)
116.9(8)	O3 Cl O5	116.8(8)
119.1(10)	O3 Cl O6	120.4(10)
117.5(11)	O4 Cl O5	117.0(11)
90.7(12)	O4 Cl O6	89.1(12)
107.7(8)	O5 Cl O6	108.2(7)
92.9(6)	H5A C5 H5B	107.6(7)
124.9(6)	H5A C5 N2	121.9(6)
108.5(7)	H5A C5 C6	109.9(7)
109.2(7)	H5B C5 N2	114.7(7)
106.9(7)	H5B C5 C6	84.7(7)
89.8(6)	H6A C6 H6B	100.8(7)
108.5(7)	H6A C6 C5	103.5(7)
109.3(7)	H6A C6 C7	116.4(7)
119.9(8)	H6B C6 C5	108.4(7)
107.7(7)	H6B C6 C7	107.9(7)
104.2(6)	H7A C7 H7B	131.3(7)
110.7(7)	H7A C7 C6	105.3(6)
103.4(6)	H7A C7 N3	98.9(6)
117.6(7)	H7B C7 C6	100.0(7)
108.8(6)	H7B C7 N3	108.9(6)
173.7(2)	O(Sol) Cu O1	173.4(3)
130.1(9)	C(Sol) O(Sol) Cu	130.6

Table 15. Least Squares Planes within the Molecule

$Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$

Atom	Deviation Å	Atom	Deviation Å
(a) Plane involving four atoms strongly coordinated to copper in Unit A: Equation: 0.53106x + 0.84339y + 0.08166z + 2.21757 = 0			
N1A	0.023	N4A	-0.022
N2A	-0.020	CuA	0.126
N3A	0.019		
(b) Plane involving four atoms strongly coordinated to copper in Unit B: Equation: -0.52996x + 0.84423y + 0.08009z + 0.43171 = 0			
N1B	-0.026	N4B	0.029
N2B	0.018	CuB	-0.128
N3B	-0.016		
(c) Plane involving the ligand (L3LH) in unit A: Equation: 0.53657x + 0.84148y + 0.06326z + 2.42805 = 0			
O1A	0.165	C4A	-0.004
O2A	0.076	C5A	0.188
N1A	0.170	C6A	-0.522
N2A	0.152	C7A	-0.166
N3A	0.141	C8A	0.183
N4A	0.076	C9A	-0.198
C1A	-0.119	C10A	-0.013
C2A	0.053	C11A	-0.032
C3A	0.073	CuA	0.261
(d) Plane involving the ligand (L3LH) in Unit B: Equation: 0.53782x - 0.84296y + 0.01251z - 0.90593 = 0			
O1B	0.098	C4B	-0.567
O2B	-0.173	C5B	0.029
N1B	0.138	C6B	-0.565
N2B	0.170	C7B	0.030
N3B	-0.060	C8B	0.215
N4B	-0.171	C9B	0.210
C1B	-0.590	C10B	0.164
C2B	-0.360	C11B	0.119
C3B	-0.305	CuB	0.147

[a]Direction cosines of the plane refer to the orgthogonal axis system a,b,c*.

[b]All atoms weighted at unity.

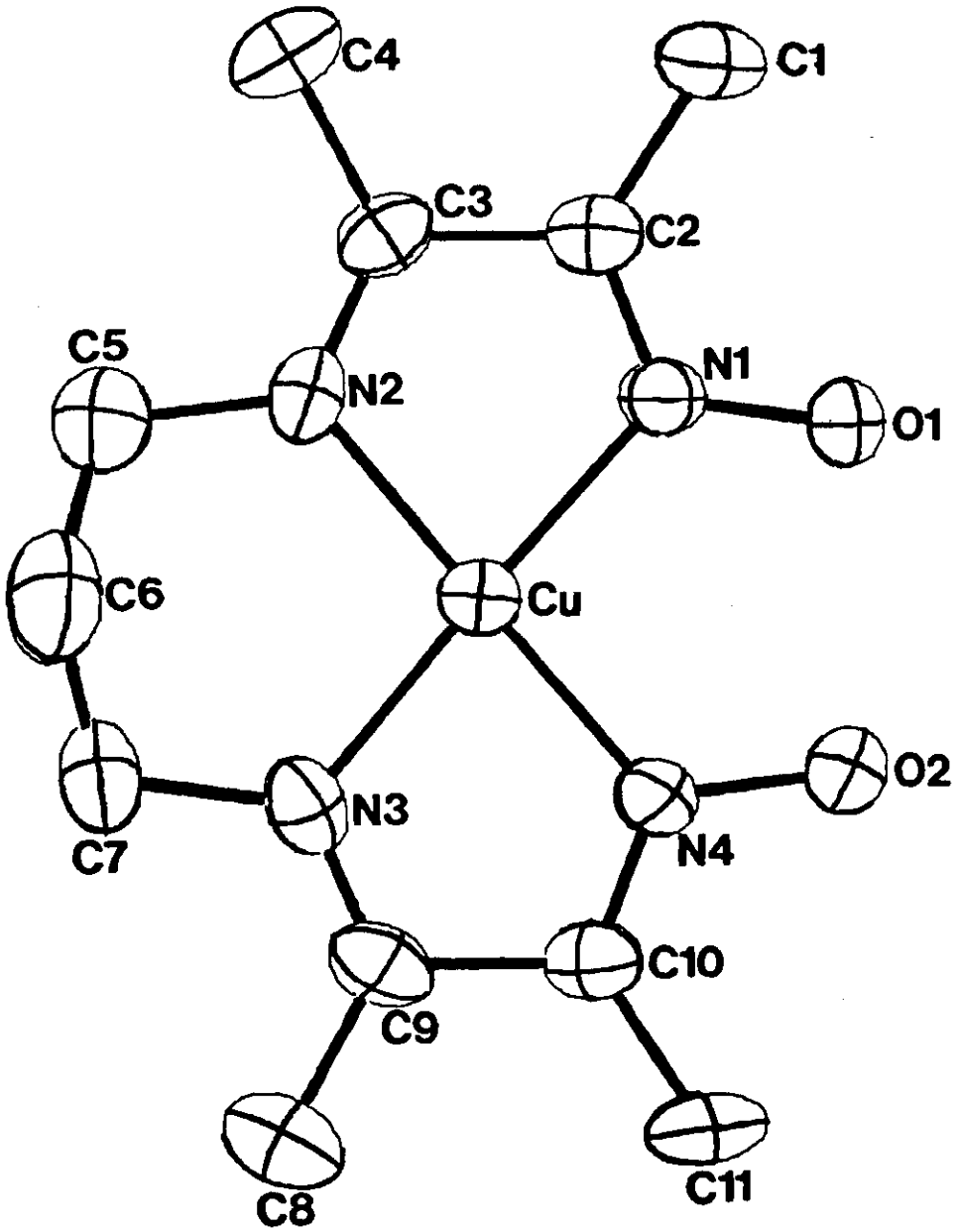


Figure 7. Monomeric Molecular Unit of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$ Omitting the Perchlorate Group and the Solvent Molecule.

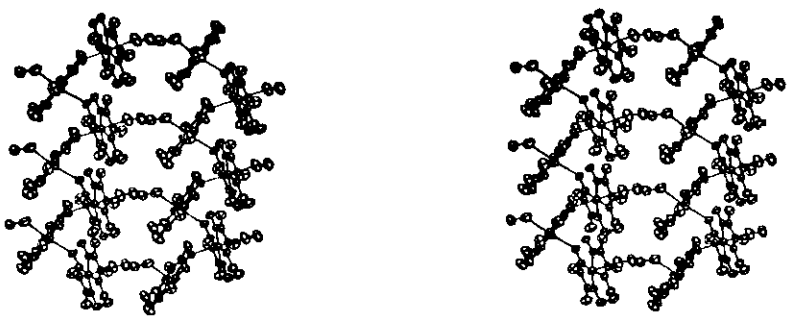
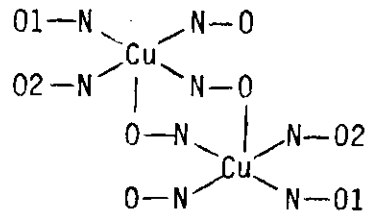


Figure 8. Stereo View of the Polynuclear Chains of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$ Omitting Perchlorate Groups.

octahedron about copper. The copper atom in each formula unit is 0.13 Å above the plane of the nitrogen atoms and there is a weak (2.492(4)Å average) axial coordination to the O1 oxime oxygen of the ligand of an adjacent formula unit. This interaction of the O1 oxime oxygen of one formula unit with the adjacent formula unit forms infinite chains with the basal coordination planes forming dihedral angles of approximately 70° with each other. The solvent molecule is more weakly coordinated (the Cu-O(sol) average distance is 2.625(10)Å) in the other axial position.

Charge balance considerations dictate the loss of one proton from the ligand (L3LH) in forming the complex. The only residual electron density near either oxime oxygen, in each formula unit, is between the two oxime oxygens and closer to the uncoordinated oxygen. The O1-O2 distances for unit A and unit B are 2.511(5) and 2.506(5) Å, respectively, which are similar to: (1) in $Cu(dmgH)_2$, as illustrated below.



$Cu(dmgH)_2$

the distance between the two oxime oxygens O1 and O2,2.562(5)Å,[7] (2) in Cu(4499)Br the oxime oxygen-oxime oxygen distance of 2.54(1)Å,[8] and

(3) in perrhenato-2-2'-(1,3-diaminopropane)bis(2-methyl-3-butanone oximato) copper(II) is 2.49Å.[31]

The most interesting feature of this structure is the association of the monomeric units to form infinite chains roughly parallel to the a-axis. The chains are separated by disordered solvent molecules and ordered perchlorate anions. The copper-oxime oxygen distances (2.493(4) and 2.491(4) Å, respectively, in units A and B) between the monomeric units indicate weak coordination. This coordination is confirmed by basal angles ranging from 84.4(2) to 101.5(2)°.

The copper-solvent oxygen distances (2.61(2) Å in both units) also may be considered a weak coordination, since in this case the basal angles range from 84.5(3) to 89.7(3)°. Due to the disorder of the solvent molecules, each solvent molecule position was assigned a multiplier of 0.50 and the copper atoms have either a square pyramidal or tetragonally distorted octahedral coordination.

The ligand (L3LH) in unit A is planar (within ±0.20 Å of the least squares plane) except for atom C6 which is 0.52 Å out of the plane. The ligand (L3LH) in unit B is much less planar with three atoms, C1, C4, and C6, deviating from the plane by more than 0.56 Å.

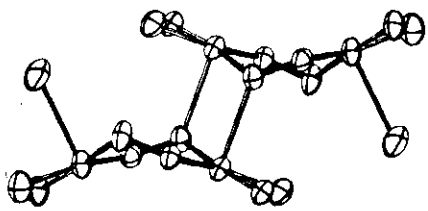
CHAPTER III

DISCUSSION

All of the single crystal x-ray diffraction structures in this study have revealed oxime bridging groups and these compounds, as well as those previously reported,[7,8] exhibit a variety of bridging arrangements and different magnetic properties.

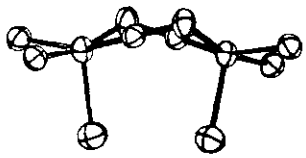
Illustrations of the coordination geometries and bridging systems of the compounds for which single crystal x-ray diffraction structures are known appear in Figure 9. One method of explaining the relationship of the magnetic properties to the molecular structures is the molecular orbital approach. In order to apply this method, various features of the structures and properties of all of these bridging oxime compounds such as magnetic behavior, metal-metal distances, geometry of the bridging arrangement, coordination of the metal ions, and relative orientation of the coordination polyhedra of the bridged metal ions will be reviewed. A summary of these features for all copper bridging oxime imine compounds appears in Table 12.

The magnetic properties of these bridging oxime complexes indicate both normal paramagnetic behavior and antiferromagnetic behavior. For $Cu(dmgH)_2$, Cu(4499)Br, and $Cu(L3LH)(ClO_4)\cdot CH_3OH$ the magnetic moments at ambient temperature range from 1.70 to 1.88 BM. The magnetic susceptibilities all follow the Curie-Weiss Equation which is normal paramagnetic behavior for copper(II) compounds. The magnetic behavior of $Cu(L2a\ell)(ClO_4)\cdot H_2O$, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, and $Cu(L2am)(ClO_4)$ is

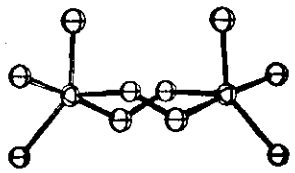


$Cu(L2am)(ClO_4)$

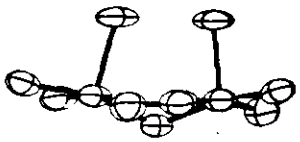
$Ni(NAH)Cl \cdot 2.5\ H_2O$



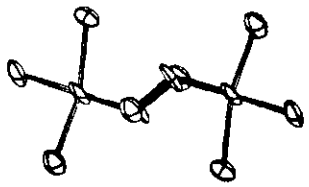
$Cu(L2pyr)(ClO_4) \cdot CH_3CN$



$Cu(4499)Br$



$[Cu_3(pyal)_3OH]SO_4 \cdot 16.3\ H_2O$



$Cu(dmgH)_2$

Figure 9. Coordination Spheres and Bridging Oxime Rings for all Polynuclear Bridging Oxime Compounds for Which Single Crystal X-Ray Diffraction Structures have been Reported.

antiferromagnetic with magnetic moments of 0.26, 0.66, and 1.38 BM, at room temperature. Detailed magnetic susceptibilities and magnetic moments are listed in Tables 1 and 2. Least squares fits for magnetic susceptibility data at various temperatures, to the Bleany and Bowers Equation:[32]

$$\chi = \frac{2g^2N\beta^2}{3kT}\left(\frac{1}{1+\frac{1}{3}\exp(J/kT)}\right) + N_\alpha$$

for $Cu(L2am)(ClO_4)$, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, and $Cu(L2pyr)(ClO_4)\cdot 2H_2O$ are presented in Table 16. The g value for $Cu(L2pyr)(ClO_4)\cdot CH_3CN$ is not within the normal range expected for these complexes. The g value was confirmed by reproducible data; deviation from the normal value may be due to impurities.

Table 16. Gyromagnetic Ratio(g), Coupling Constant (J), and Temperature Independent Paramagnetism ($N\alpha$) from a Least Squares Fit to the Bleany and Bower Equation for $Cu(L2am)(ClO_4)$, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, and $Cu(L2pyr)(ClO_4)\cdot 2H_2O$.

Complex	g	J	$N\alpha$
$Cu(L2am)(ClO_4)$	2.13	-293	9
$Cu(L2pyr)(ClO_4)\cdot CH_3CN$	1.59	-599	87
$Cu(L2pyr)(ClO_4)\cdot 2H_2O$	2.08	-582	158

Of several structural features which may affect the magnetic properties, the first to be considered is the metal-metal distance. Copper-copper distances in these compounds range from 3.268(2)Å in the four-membered ring of $Cu(L2am)(ClO_4)$ to 5.129(4)Å in $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$. Such distances are generally considered too long for significant metal-metal interactions and direct exchange coupling.[33] In fact a detailed magnetic and molecular orbital study of the copper(II) acetate monohydrate dimer, in which the Cu-Cu distance is 2.5-2.7 Å, has shown that metal-metal bonding contributes very little to the stability of the dimer.[34]

Studies comparing the copper-copper distances and magnetic moments of similar formates and acetates[35] and of di-μ-hydroxy complexes[36] of copper have shown antiferromagnetic coupling which increases with increasing metal-metal distance. Several complexes with metal-metal distances in the 5 to 6 Å range are reported to have significant magnetic coupling through polyatomic bridges.[37,39] Since a metal-metal interaction and direct exchange coupling are assumed to be negligible, coupling by superexchange through a sigma or pi pathway must be considered in order to explain the antiferromagnetic properties of these compounds. To discuss the pi pathway of superexchange it is necessary to describe the geometry of the bridging oxime rings.

In most of the compounds studied, the bridging oxime groups form cyclic systems involving metal atoms. These rings fall into four categories: six-membered planar, six-membered chair, six-membered boat, and four-membered planar. The compound $Cu(L2a\ell)(ClO_4)\cdot H_2O$ forms a six-membered planar ring, and $Cu(dmgH)_2$ forms a six-membered ring with a chair conformation. The dimeric units of Cu(4499)Br, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$,

and Cu(L2am)(ClO_4), form six-membered rings with a twisted boat conformation. The association of the dimers in Cu(L2am)(ClO_4) forms planar four-membered rings.

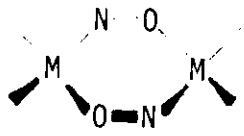
Exchange coupling by a pi mechanism must be considered for the planar six-membered ring compounds, since in a planar ring system the d_{xz} and d_{yz} orbitals of copper and the p-orbitals of oxygen and nitrogen are favorably aligned for pi overlap. Such an explanation was suggested in the case of four-membered ring compounds. The explanation related the magnitude of the antiferromagnetic coupling constant to the ring angle at copper. In these four-membered ring compounds, in which the angle was considerably less than 90°, the coupling decreased with increasing angle. In the only planar six-membered ring compound Cu(L2aℓ)(ClO_4)·H_2O this angle (108.3(2)°) is considerably greater than 90°. The explanation requires a pi type molecular orbital, involving d_{xz} or d_{yz} orbitals, of higher energy than the sigma type molecular orbital, which involved the d_{xy} orbital. There is no evidence for this order of energies.

A planar six-membered ring would provide a pi pathway, but the overlap of the oxime oxygen and nitrogen sigma orbitals with the half-filled copper d_{xy}(or $d_{x^2-y^2}$, depending on the orientation of the axes) orbital would also provide a sigma pathway. The antiferromagnetic coupling in the six-membered rings of Cu(L2aℓ)(ClO_4)·H_2O, Cu(L2pyr)(ClO_4)·CH_3CN, and Cu(L2am)(ClO_4), indicates that significant coupling occurs for non-planar rings as well as planar rings. The contribution of a pi pathway to the coupling by superexchange in these compounds is small, because of the alignment of the orbitals. For this

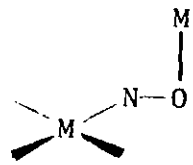
reason further discussion will concentrate on explaining the magnetic coupling by superexchange through a sigma pathway.

For the polyatomic bridged compound of copper(II) and the diimine of 2,4-pentanedione and 2-aminoethanol,[37] in which there are O-H-O bridges, exchange almost certainly occurs by a sigma pathway since hydrogen has no orbitals that are appropriate for pi bonding. In addition, recent correlations[36] between the angle at oxygen for dimeric copper-hydroxide complexes with both planar and bent four-membered Cu_2O_2 rings provide evidence for a sigma pathway. A molecular orbital approach to the coupling by a sigma pathway will begin with a discussion of the bridging arrangements, the coordination arrangement of the copper atoms in these compounds, and the orientation of the half-filled metal d-orbitals in the polynuclear units.

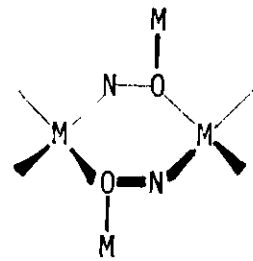
There are three distinct types of oxime bridges in these copper oxime complexes. In type (A), the M-O bond is in, or only slightly out of, the plane defined by O-N-M (the sp^2 plane of the oxime nitrogen). In type (B) the M-O bond is perpendicular to the sp^2 plane of the oxime nitrogen. In type (C), two metals are coordinated to the bridging oxygen--one M-O bond is in the sp^2 plane of the nitrogen and the other perpendicular to this sp^2 plane.



(A)



(B)



(C)

Of the structures studied, $Cu(L2a\ell)(ClO_4)\cdot H_2O$ and $Cu(L2pyr)(ClO_4)\cdot CH_3CN$ have type (A) bridges; $Cu(dmgH)_2$, Cu(4499)Br, and $Cu(L3LH)(ClO_4)\cdot \frac{1}{2}CH_3OH$, have type (B) bridges; and $Cu(L2am)(ClO_4)$ has type (C) bridges.

In a d^9 configuration with only one half-filled orbital, the coordination arrangement and the orientation of the half-filled orbital with respect to the coordination arrangement become important in explaining magnetic coupling in a polynuclear compound. The metal atoms in these oxime bridged compounds have three different coordination arrangements. The $Cu(L2a\ell)(ClO_4)\cdot H_2O$ dimer has both copper atoms in a tetragonally distorted octahedral coordination. Due to disorder of the methanol molecule in $Cu(L3LH)(ClO_4)\cdot \frac{1}{2}CH_3OH$, half of the copper atoms have tetragonally distorted octahedral coordination and the other half of the copper atoms have square pyramidal coordination. In $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, $Cu(L2am)(ClO_4)$ and $Cu(dmgH)_2$ all copper atoms have square pyramidal coordination with a weak axial coordination. In Cu(4499)Br the copper atoms have trigonal bipyramidal coordination.

In addition, the relative orientation of the coordination polyhedra, including the atomic orbital with the unpaired electron, is necessary to explain magnetic properties by a molecular orbital approach. Figure 9 shows the orientation of coordination polyhedra in all bridging oxime complexes for which single crystal x-ray diffraction structures have been determined. In $Cu(L2a\ell)(ClO_4)\cdot H_2O$ the basal planes of the two copper atoms are coplanar with the oxime bridging groups. For $Cu(L2pyr)(ClO_4)\cdot CH_3CN$ and $Cu(L2am)(ClO_4)$ the basal planes of the two copper atoms in the dimeric units are twisted and bent toward each other to form the twist boat six-membered ring. The two basal planes

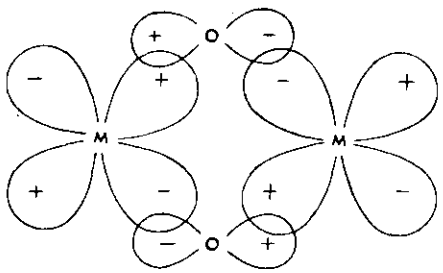
of adjacent copper atoms in the zig-zag chain of $Cu(L3LH)(ClO_4)\cdot\frac{1}{2}CH_3OH$ form a dihedral angle of approximately eighty degrees. In $Cu(dmgH)_2$ the basal planes of the two copper atoms in the dimer are parallel but separated by 2.3 Å with a copper-copper distance of 3.851 Å. The trigonal bipyramidal coordination polyhedra of the copper atoms in Cu(4499)Br are oriented such that the six-membered bridging oxime ring is formed by copper atoms, axial oxime nitrogen atoms, and equatorial oxime oxygen atoms. The two N-Cu-O-N bonding sequences form planes which intersect at an angle of approximately 110° to form the boat conformation ring.

In polynuclear copper(II) complexes normal paramagnetism is expected for structures in which half-filled metal orbitals are not mixed in forming molecular orbitals. Magnetic coupling is expected when the half-filled metal orbitals are mixed with filled orbitals of the bridging ligand orbitals in forming molecular orbitals. When this interaction results in antibonding orbitals which are equal in energy (e.g. Figure 2c) ferromagnetism occurs. Antiferromagnetism results when the energy difference between these antibonding orbitals is sufficient to cause pairing in the lower lying orbital (e.g. Figure 2b).

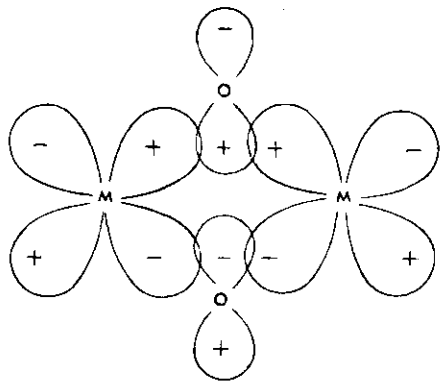
The molecular orbital approach may be used to explain the magnetic coupling through a sigma pathway. Bertrand and Eller[2] have made a qualitative study of the molecular orbitals of the di-μ-hydroxo copper(II) compounds for which MacGregor et al.[36] report magnetic coupling which varies with the Cu-O-Cu angle. Hay et al.[45] present a detailed quantitative treatment of the molecular orbitals of these and chlorine bridged copper(II) systems. Hay et al. relate molecular orbitals to magnetic coupling treatments presented by Goodenough,[46,47] Anderson,[48]

Kanamori,[49] and Ginsberg.[33] Both the qualitative and the quantitative studies describe in great detail the molecular orbital approach to the explanation of magnetic coupling through superexchange. Ideas from these studies will be employed in the explanation of the magnetic coupling in six-membered bridging oxime rings.

In square planar or square pyramidal copper(II) dimers the unpaired electron on each copper would be in the d_{xy} orbital (or $d_{x^2-y^2}$ orbital depending upon the orientation of the axes). Assuming C_{2h} symmetry these two half-filled d_{xy} orbitals may be combined into linear combinations which transform with A_g and B_u symmetries and these will interact with filled s and p orbitals on the bridging atoms. The symmetric linear combination, A_g, overlaps the p_x orbitals and the asymmetric linear combination, B_u, overlaps the p_y orbitals.



A_g



B_u

At a Cu-O-Cu angle of 90° the orbital overlap in both cases would be identical and the two molecular orbitals would have equal energies. A change in the Cu-O-Cu angle would change the overlap and destroy the accidental degeneracy. When the s-orbitals of the bridging atoms are

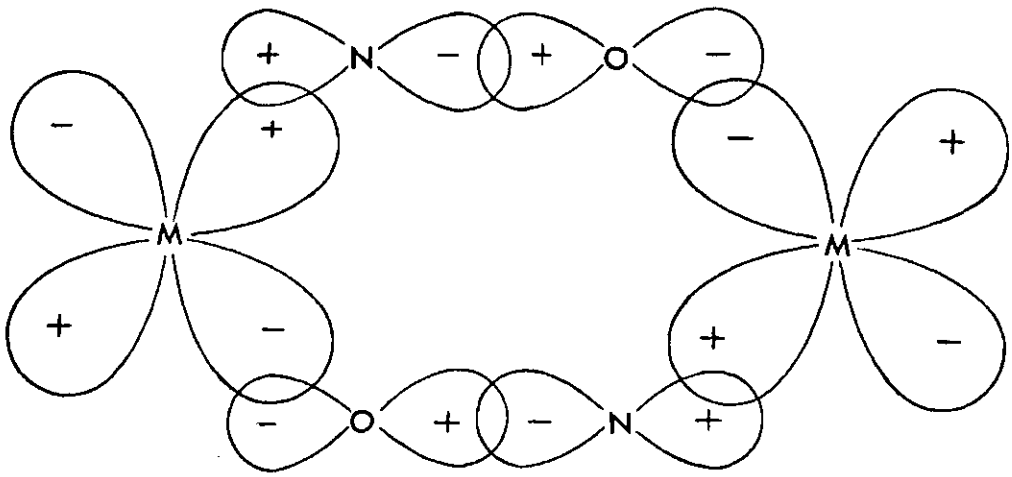
considered these atomic orbitals would mix into the B_u molecular orbitals and the overlap at 90° would not be the same for A_g and B_u. The experimental results of MacGregor et al.[36] in which coupling constants of ferromagnetic and antiferromagnetic compounds were compared to the Cu-O-Cu angle, show that the accidental degeneracy occurs at an angle of approximately 97°. Both qualitative[2] and quantitative[45] considerations of the energy levels for these complexes indicate that ferromagnetic behavior would be expected for angles of about 97°, with antiferromagnetic coupling occurring at angles both much larger and much smaller. But this prediction cannot be completely tested since no compound with an angle of less than 95° has been reported.

There are also two examples of polynuclear copper(II) compounds with four-membered Cu_2O_2 rings in which the coordination about copper is square pyramidal and the bridging involves axial and basal positions. The observed ferromagnetic coupling can be explained since the angle at copper within each of the four-membered rings deviates significantly from 90° so that the d_{xy} and d_z^2 orbitals can mix. This situation is similar to the $[(Cl_4\text{-}Cu)\text{-}Cl\text{-}(CuCl_4)]^{5-}$ case considered by Hay et al.[45] where the coupling is explained by a mixing of the d_{xy} and d_z^2 orbitals as the symmetry of the $CuCl_5^{3-}$ fragment goes from C_{4v} to C_{2v}. In the four-membered Cu_2O_2 ring the mixing of these orbitals and bridging atoms s-orbitals into molecular results in identical overlap for the symmetric and asymmetric combinations and thus identical energies.

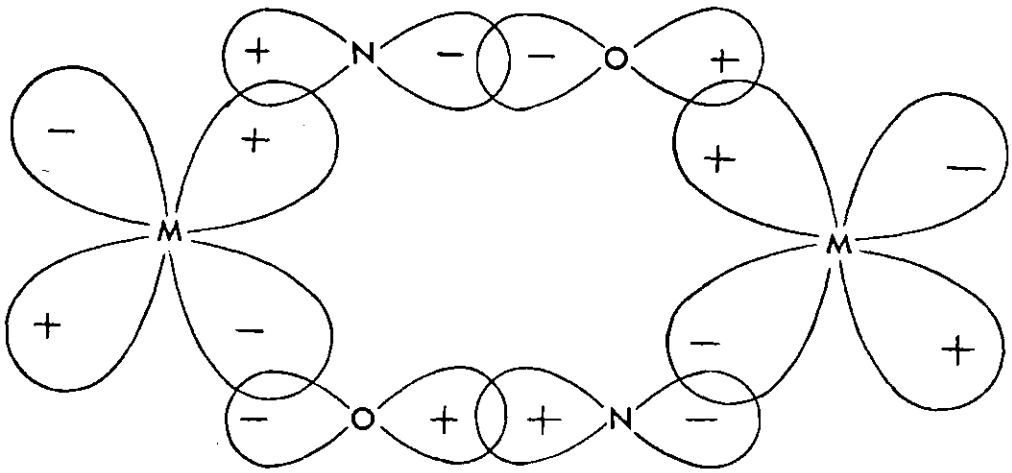
Magnetic coupling through polyatomic bridges is also known.[38,39,44] For a six-membered $Cu_2(NO)_2$ ring with C_2 symmetry the symmetric linear combinations of the half-filled d_{xy} orbitals interact with the symmetric

antibonding nitrogen and oxygen molecular orbital. The asymmetric linear combination interacts with the asymmetric bonding nitrogen and oxygen molecular orbital to give the molecular orbitals in Figure 10. An energy-level diagram appears in Figure 11. The energy difference of the orbitals labeled B_u and A_g allows the electrons from the d_{xy} orbitals to pair in the A_g orbitals to give a singlet ground state and antiferromagnetic coupling.

In contrast to the four-membered rings which have both linear combinations of d_{xy} orbitals interacting with non-bonding bridging atomic orbitals, the six-membered rings have linear combinations of d_{xy} orbitals interacting with bonding and antibonding bridging atom molecular orbitals. This difference should result in a larger energy separation and less variation of magnetic properties with structural parameters.



A_g



B_U

Figure 10. Combinations of Metal and Bridging Atom Orbitals in Molecular Orbital Treatment of σ Coupling.

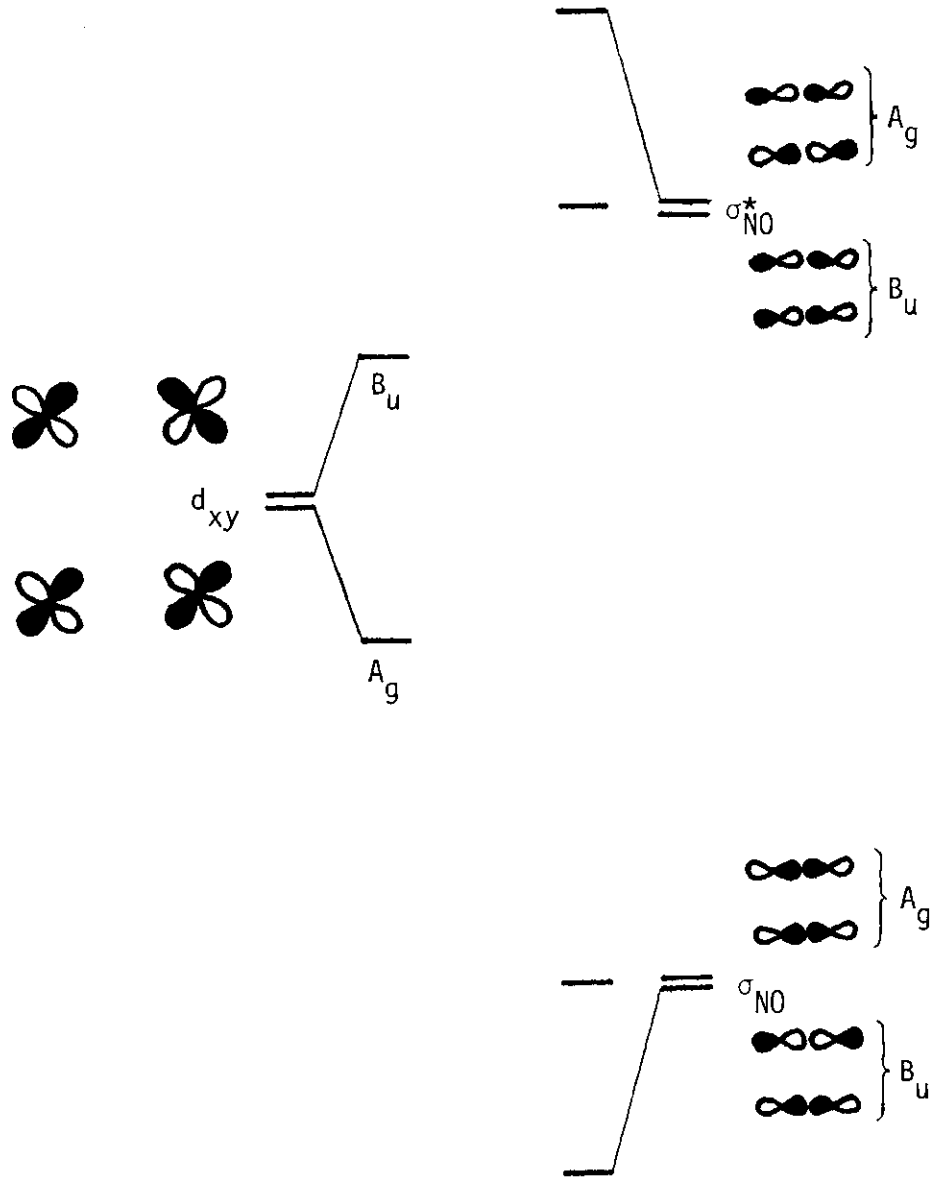


Figure 11. Energy Level Diagram for Molecular Orbitals of Six-Membered $Cu_2(NO)_2$ Rings.

CHAPTER IV

CONCLUSION

The information obtained from the single crystal x-ray diffraction structures in this systematic study of copper(II) oxime imines, when combined with existing single crystal x-ray diffraction structural information of copper(II) oximes, clarifies the relationship of the magnetic properties of these compounds to their molecular structure.

Since all of these compounds have relatively long metal-metal distances, magnetic coupling from direct exchange does not appear probable. Therefore all magnetic coupling is assumed to be derived from superexchange through the oxime bridging systems.

The occurrence of antiferromagnetic coupling can be based on molecular structure as interpreted by molecular orbital considerations. According to a molecular orbital approach compounds with two half-filled metal d orbitals overlapping the same bridging group p orbitals should exhibit antiferromagnetic coupling. In this study, $Cu(L2a\ell)(ClO_4)\cdot H_2O$, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, and $Cu(L2am)(ClO_4)$ show the kind of structure, which allows these overlaps, and all three compounds show antiferromagnetic behavior.

Three compounds, $Cu(L3LH)(ClO_4)\cdot \frac{1}{2}CH_3OH$, $Cu(dmgH)_2$) and Cu(4499)Br, have molecular structures in which the half-filled metal d orbitals overlap filled p orbitals of bridging atoms and filled metal d orbitals overlap orthogonal p orbitals on these same bridging atoms. The molecular orbital approach correctly predicts that these compounds exhibit normal

paramagnetism since the O-Cu-O angles are all approximately 90° (see Table 12) and no mixing of the half-filled metal d-orbitals occurs. The four-membered Cu_2O_2 ring in the Cu(L2am)(ClO_4) tetramer with an O-Cu-O angle of 93.1(2)° should exhibit normal paramagnetic behavior as well.

Although cyclic polyatomic bridging compounds have been reported[38,39] to have ferromagnetic coupling, these have been for extended pi bonded bridging groups with nickel(II). No ferromagnetic copper(II) oximes have been reported even though reasonable structures for ferromagnetic coupling can be suggested on the basis of molecular orbital theory.

From existing information the distinction between the magnetic moments of Cu(L2aℓ)(ClO_4)·(H_2O), Cu(L2pyr)(ClO_4)·CH_3CN and Cu(L2am)(ClO_4) can be explained only by the observation that increased deviation from planarity of the bridging oxime ring results in a higher magnetic moment at room temperature. The deviation from planarity of the six-membered ring may decrease orbital overlap and hence coupling and superexchange. Caution must be observed before drawing such conclusions in complexes with low magnet moments, however. First, measurements involve very small weight changes, and second small amounts of paramagnetic impurities may make significant contributions to the magnetic susceptibilities, especially at low temperatures.

A single crystal x-ray diffraction structure of the dihydrate or unsolvated complex of Cu(L2pyr)(ClO_4), each having room temperature magnetic moments of approximately 0.9 BM, would provide assistance in clarifying the structural relationship to the magnetic properties of these complexes; unfortunately, suitable single crystals have eluded preparation.

APPENDIX

OBSERVED (FO) AND CALCULATED (FC) STRUCTURE FACTORS FOR $Cu(L2a\ell)(ClO_4)\cdot H_2O$, $Cu(L2pyr)(ClO_4)\cdot CH_3CN$, $Cu(L2am)(ClO_4)$, AND $Cu(L3LH)(ClO_4)\cdot \frac{1}{2}CH_3OH$

$Cu(L2a\ell)(ClO_4)\cdot H_2O$

H	K	FO	FC
	L=-13		
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4	1	13	13
5	1	27	26
4	2	11	10
5	2	16	15
2	3	28	28
4	3	23	21
5	3	34	32
3	4	15	14
	L=-12		
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2	1	21	20
3	1	12	9
4	1	15	16
5	1	8	8
6	1	11	13
1	2	21	20
2	2	35	35
3	2	19	18
5	2	16	13
6	2	34	34
7	2	22	23
1	3	16	17
2	3	21	22
4	3	16	17
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2	4	31	32
3	4	14	13
4	4	13	11
5	4	13	13
6	4	43	42
1	5	12	7
6	5	12	11
1	6	18	17
2	6	37	38
3	6	29	29
5	6	30	30
4	7	13	9
	L=-11		
1	1	12	9
2	1	15	17
3	1	20	20
5	1	23	23
6	1	12	12
7	1	49	48
1	2	18	19
4	2	16	15
5	2	9	9
6	2	14	15
7	2	9	6
1	3	9	9
2	3	31	31
3	3	61	58
4	3	37	37
6	3	35	36
7	3	26	24
4	4	18	16
5	4	8	10
6	4	9	12
7	4	8	7
1	5	16	17
2	5	40	40
3	5	46	49
4	5	28	27
5	5	10	10
6	5	30	30
7	5	22	24
1	6	8	2
3	6	20	20
4	6	8	4
5	6	21	21
1	7	16	17
2	7	43	44
3	7	27	26
4	7	14	12
6	7	22	20
1	8	8	5
5	8	8	4
2	9	22	23
3	9	33	34
4	9	19	17
	L=-10		
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2	1	11	15
4	1	12	11
5	1	22	23
7	1	15	20
1	2	18	17
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3	2	34	37
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8	4	40	41
1	5	26	26
3	5	16	14
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7	5	13	12
1	6	18	18
3	6	46	48
4	6	56	55
5	6	21	21
6	6	14	14
7	6	23	24
1	7	8	8
2	7	8	6
3	7	16	14
5	7	13	11
1	8	18	17
2	8	9	7
3	8	33	36
4	8	44	45
5	8	23	22
6	8	15	16
1	9	11	13
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2	10	18	17
3	10	25	24
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5	10	15	18
2	11	13	9
3	11	11	8
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3	6	18	18
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6	6	12	13
1	7	44	44
3	7	32	31
4	7	16	14
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2	8	19	21
4	8	8	6
5	8	9	9
7	8	13	12
1	9	29	29
2	9	12	11
4	9	33	33
5	9	43	42
6	9	15	12
2	10	22	20
3	10	16	19
4	10	12	10
5	10	8	4
1	11	19	19
2	11	23	21
4	11	34	34
5	11	39	39
1	12	10	10
3	12	21	22
4	12	10	14
	L= -8		
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3	1	12	13
4	1	30	29
5	1	17	19

H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC
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4	2	54	56	5	12	34	33	1	8	27	28	2	4	85	81
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8	2	20	18		L=	-7		5	8	22	21	6	4	20	23
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2	2	58	59
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2	6	9	14
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3	7	21	21
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2	13	11	10
1	14	31	33
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2	3	82	81
3	3	40	39
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1	4	13	12
2	4	23	25
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1	6	8	7
2	6	35	36
3	6	43	42
4	6	21	19
1	7	8	3
0	8	22	19
2	8	21	22

H	K	FO	FC
3	8	40	39
0	9	9	9
1	9	13	16
2	9	8	5
3	9	15	14
0	10	28	28
1	10	16	17
2	10	24	24
0	11	22	24
1	11	7	4
0	12	14	20

L= 9

H	K	FO	FC
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0	2	28	26
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2	2	14	14
0	3	27	28
1	3	34	34
2	3	13	11
3	3	35	35
0	4	14	12
0	5	41	41
1	5	22	21
2	5	8	12
3	5	20	21
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2	6	6	11
0	7	47	48
1	7	11	10
2	7	12	12
0	8	25	28
1	8	26	27
0	9	31	30
0	10	25	26
1	10	15	13
0	11	22	22

L= 10

H	K	FO	FC
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1	0	27	25
2	0	23	23
3	0	19	20
1	1	18	18
3	1	9	6
0	2	32	34
1	2	33	34
2	2	9	6
1	3	8	9
0	4	39	40

H	K	FO	FC
1	4	36	36
1	5	26	27
2	5	9	9
0	6	26	25
1	6	35	34
2	6	18	19
0	7	8	1
1	7	13	13
0	8	28	29
1	8	32	31
0	9	9	11
0	10	31	32

L= 11

H	K	FO	FC
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2	1	18	16
0	2	17	19
2	2	15	14
0	3	17	18
1	3	21	20
1	4	9	1
0	5	13	11
1	5	33	34
0	6	9	10
1	6	11	10
0	7	20	21

L= 12

H	K	FO	FC
0	3	9	7

$Cu(L2pyr)(ClO_4)\cdot CH_3CN$

K	L	FO	FC
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0	10	19	18
0	12	64	70
0	16	12	12
0	18	8	8
2	2	98	77
2	3	20	24
2	4	19	27
2	5	16	22
2	6	99	112
2	7	113	115
2	8	48	50
2	9	25	26
2	10	8	9
2	11	69	72
2	12	32	31
2	13	25	25
2	14	7	6
2	16	5	3
2	19	6	6
4	0	100	97
4	1	139	133
4	2	129	129
4	3	165	162
4	4	77	77
4	5	120	125
4	6	12	10
4	7	110	114
4	9	49	53
4	10	58	60
4	11	48	48
4	12	13	15
4	14	7	4
4	15	11	13
4	18	12	12
6	0	67	68
6	1	23	22
6	2	110	104
6	3	10	9
6	4	114	118
6	6	33	34
6	7	10	10
6	8	37	37
6	10	47	45
6	11	17	16
6	13	8	7
6	16	10	12
8	0	15	17
8	1	47	47
8	2	51	52
8	3	68	70
8	4	37	36
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8	7	26	25
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8	9	30	29
8	10	19	19
8	11	6	5
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10	2	7	6
10	3	25	26
10	4	17	16
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10	6	14	17
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0	8	72	84
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0	12	43	47
0	14	40	39
0	16	21	20
0	18	22	21
1	0	196	199
1	1	156	152
1	2	224	236
1	3	17	19
1	4	185	180
1	5	8	9
1	6	70	67
1	7	12	14
1	8	84	93
1	9	54	55
1	10	87	90
1	11	56	60
1	12	11	9
1	13	27	29
1	14	6	5
1	16	8	9
1	18	8	6
1	19	9	9
2	1	7	2
2	2	240	230
2	3	113	110
2	4	73	78
2	5	55	51
2	6	61	56
2	7	13	14
2	8	52	57
2	9	42	45
2	10	23	25
2	11	37	39
2	12	8	8
2	13	50	50
2	14	18	18
2	15	29	30
2	17	24	24
2	18	7	5
2	19	21	20
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3	2	8	15
3	3	154	143
3	5	111	109
3	6	14	10
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3	8	26	28
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3	12	52	54
3	13	8	8
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3	15	9	7
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4	16	16	17
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4	18	10	10
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6	16	18	19
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7	1	54	56
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0	12	68	73
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0	16	9	7
0	18	6	7
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1	2	90	86

K	L	FO	FC	K	L	FO	FC	K	L	FO	FC	K	L	FO	FC
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1	5	77	77	4	7	74	74	8	3	39	39	2	5	24	23
1	6	20	24	4	8	17	17	8	4	10	11	2	6	70	70
1	7	49	52	4	9	39	38	8	5	37	36	2	7	129	127
1	8	23	21	4	10	36	38	8	6	15	15	2	8	66	70
1	9	38	39	4	11	18	19	8	7	21	20	2	9	125	129
1	10	75	78	4	12	34	35	8	8	13	12	2	10	47	47
1	11	50	53	4	13	6	3	8	9	20	20	2	11	87	91
1	12	90	92	4	15	6	7	9	1	12	14	2	12	41	44
1	13	43	43	4	16	9	11	9	2	16	14	2	13	82	82
1	14	5	6	5	1	12	7	9	3	10	7	2	14	40	40
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1	16	45	46	5	3	43	44	9	5	9	7	2	16	21	20
1	17	25	24	5	4	19	18	9	6	12	12	2	17	39	39
1	18	46	45	5	5	32	32	9	7	27	29	2	18	20	21
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2	11	65	65	6	1	20	18	0	4	118	116	3	11	18	16
2	12	14	15	6	2	30	29	0	6	35	30	3	13	11	7
2	13	30	31	6	3	14	14	0	8	145	150	3	15	10	9
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2	15	8	7	6	5	5	4	0	12	94	99	4	1	34	34
2	19	10	6	6	6	43	41	0	14	84	84	4	3	52	54
3	1	123	114	6	7	15	14	0	16	53	51	4	4	33	36
3	2	12	7	6	8	50	51	0	18	45	45	4	5	83	78
3	3	35	33	6	10	28	28	1	0	87	81	4	6	24	21
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3	7	7	5	6	15	8	8	1	4	64	61	4	10	35	31
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3	9	42	44	7	1	25	27	1	6	53	55	4	12	49	50
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7	14	8	7
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9	5	10	10
9	8	8	5
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H= 4

K	L	FO	FC
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0	6	136	136
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0	12	10	9
0	14	12	11
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0	18	9	12
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1	2	78	64
1	3	109	98
1	4	23	14
1	5	57	61
1	6	94	96
1	7	124	119
1	8	97	96
1	9	82	86
1	10	37	38
1	11	53	57
1	12	76	77
1	13	46	47
1	14	71	70
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1	16	37	38
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1	19	17	15
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2	3	71	71
2	4	19	23
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H= 5

K	L	FO	FC
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0	12	79	82
0	14	26	26
0	16	13	12
0	18	21	20
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1	2	76	74
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1	4	147	147
1	5	42	44
1	6	103	102
1	7	62	66
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1	10	73	74
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1	15	26	26
1	16	19	19
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2	7	95	90
2	8	16	16
2	9	52	53

K	L	FO	FC
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2	12	21	22
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2	14	7	6
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2	16	23	23
2	17	11	10
2	18	11	11
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3	2	11	16
3	3	89	89
3	4	25	21
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3	8	19	18
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H= 6

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K	L	FO	FC
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K	L	FO	FC
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5	10	42	42
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5	16	24	23
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6	15	9	5
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7	6	24	23
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7	9	25	25
7	10	25	24
7	11	18	19
7	12	29	29
7	13	14	12
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8	4	20	19
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8	6	11	9
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8	11	11	7
9	1	7	11
9	2	10	7
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9	5	11	10
9	6	7	9
9	7	15	17
9	8	13	14
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10	2	12	11

H= 9

K	L	FO	FC
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0	8	19	20
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0	12	55	57
0	14	17	16
0	16	53	55
0	18	54	53
1	0	17	18
1	1	4	3
1	2	46	48
1	3	7	10
1	4	31	29
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1	6	26	25
1	7	4	2
1	8	7	9
1	10	12	10
1	11	11	10
1	12	6	7
1	14	6	4
2	1	7	0
2	2	6	4
2	3	66	66
2	4	43	43
2	5	83	85
2	6	61	61
2	7	61	60
2	8	27	26
2	9	59	59
2	10	45	47
2	11	67	66
2	12	37	37
2	13	39	39
2	15	46	47
2	16	19	19
2	17	51	50
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K	L	FO	FC
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4	8	82	77
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4	11	38	37
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4	13	39	39
4	14	32	32
4	15	32	32
4	16	18	19
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5	2	37	35
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6	8	77	77
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8	8	15	15
8	9	20	22
8	11	22	20
9	1	9	12
9	3	15	15
9	4	7	6

K	L	FO	FC
9	8	7	3

H= 10

K	L	FO	FC
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0	8	63	64
0	10	42	42
0	12	5	1
1	1	6	5
1	2	16	20
1	3	14	17
1	4	6	7
1	5	58	57
1	6	70	65
1	7	87	84
1	8	84	81
1	9	43	44
1	10	41	40
1	11	27	27
1	12	33	32
1	13	28	27
1	14	70	72
1	15	34	34
1	16	43	43
1	17	27	26
1	18	34	31
2	0	48	43
2	1	22	20
2	2	76	74
2	3	23	24
2	4	21	21
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2	7	57	55
2	8	22	24
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2	10	29	27
2	11	29	31
2	12	22	23
2	13	6	0
2	14	7	8
2	16	10	10
2	17	8	3
3	1	23	23
3	2	69	66
3	4	31	31
3	5	32	30
3	6	29	28
3	7	90	89
3	8	42	42
3	9	50	50
3	11	61	61
3	12	9	4
3	13	63	64

K	L	FO	FC
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3	17	40	39
4	1	32	32
4	2	5	4
4	3	6	7
4	5	13	9
4	6	26	26
4	7	35	36
4	9	25	24
4	10	14	14
4	11	19	19
4	12	14	14
4	15	9	7
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5	2	38	36
5	3	7	5
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5	7	38	39
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5	10	53	51
5	12	54	56
5	13	27	28
5	14	32	31
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6	3	6	3
6	5	15	13
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9	3	12	10
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9	7	15	15

H= 11

K	L	FO	FC
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0	6	42	43
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0	10	55	57
0	12	36	35
0	14	61	60
0	16	53	53
1	0	174	172
1	1	43	47
1	2	74	76
1	3	40	35
1	4	46	41
1	5	61	62
1	6	95	96
1	7	28	29
1	8	36	39
1	9	35	36
1	10	11	9
1	11	10	10
1	12	37	37
1	14	8	7
1	16	7	3
2	1	60	56
2	2	17	19
2	3	13	11
2	4	26	25
2	5	47	47
2	6	30	30
2	7	66	66
2	8	32	33
2	9	52	53
2	10	45	44
2	11	51	51
2	12	43	43
2	13	39	38
2	14	19	18
2	15	43	43
2	16	14	15
2	17	36	34
3	0	62	62
3	1	103	106
3	2	9	9
3	3	62	58
3	4	45	45
3	5	80	82
3	6	31	30
3	7	57	56
3	8	20	21
3	9	21	20
3	10	35	34
3	11	42	43

K	L	FO	FC
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3	14	11	12
3	15	6	6
4	1	10	10
4	2	17	17
4	3	16	13
4	5	29	30
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4	7	39	39
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4	11	46	45
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4	13	30	30
4	14	9	10
4	15	19	19
4	16	14	12
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5	2	82	81
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5	8	11	10
5	9	9	10
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6	3	9	7
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6	8	35	34
6	10	36	36
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6	14	35	35
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7	1	23	24
7	2	39	40
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7	4	55	54
7	5	16	17
7	6	10	9
7	7	6	3
7	8	6	4
7	9	16	17
7	10	16	18
7	11	14	13

K	L	FO	FC
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8	5	13	10
8	6	12	10
8	7	11	11
8	8	7	5
8	9	12	11
9	0	21	21
9	1	19	20
9	3	26	26
9	4	7	5
9	5	21	23

H= 12

K	L	FO	FC
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0	4	138	138
0	6	68	70
0	8	81	82
0	10	77	77
0	12	10	11
0	16	16	15
1	1	11	17
1	2	60	56
1	3	19	18
1	4	31	35
1	5	46	46
1	6	35	38
1	7	23	22
1	8	16	18
1	9	28	29
1	10	24	26
1	11	12	12
1	12	6	6
1	14	7	8
1	15	9	7
1	16	18	18
1	17	10	10
2	1	113	112
2	2	63	63
2	3	117	114
2	4	42	45
2	5	84	86
2	6	33	33
2	7	46	44
2	9	58	58
2	10	36	36
2	11	11	10
2	12	10	11
2	14	21	21
2	16	9	9
3	1	21	21
3	2	15	16
3	3	16	18
3	4	29	33

K	L	FO	FC
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3	6	24	23
3	7	8	5
3	8	14	13
3	9	20	20
3	11	17	17
3	12	18	18
3	13	7	9
3	15	19	19
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4	1	91	92
4	2	59	62
4	3	108	108
4	4	26	26
4	5	72	71
4	6	81	78
4	7	20	21
4	8	23	22
4	9	11	10
4	11	28	27
4	12	13	13
4	13	24	24
4	15	11	11
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5	2	19	18
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5	4	21	20
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5	7	11	10
5	8	23	23
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5	14	17	17
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6	2	60	61
6	3	12	9
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6	5	9	9
6	6	58	57
6	7	17	16
6	8	17	18
6	10	22	20
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7	2	25	25
7	3	7	7
7	4	10	8
7	5	13	13
7	6	13	13
7	8	16	16
8	0	12	9
8	1	62	62
8	2	7	4
8	3	29	29
8	4	16	16
8	5	38	39

K	L	FO	FC
8	6	23	23
8	7	22	23
8	8	16	14
8	9	15	13
9	1	8	5
9	2	13	13
9	3	10	9
9	5	7	6

H= 13

K	L	FO	FC
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0	4	25	29
0	6	36	36
0	10	21	18
0	12	37	38
0	14	13	12
0	16	18	16
1	0	57	59
1	1	33	36
1	2	102	103
1	3	60	56
1	4	134	131
1	5	61	61
1	6	88	83
1	7	30	29
1	8	46	47
1	9	46	48
1	10	39	38
1	12	31	32
2	2	46	47
2	3	18	21
2	4	21	20
2	5	13	14
2	7	15	15
2	8	40	42
2	9	11	13
2	10	31	32
2	11	8	6
2	12	21	22
2	13	19	20
2	15	11	11
2	16	12	12
3	0	18	17
3	1	112	113
3	2	25	22
3	3	122	120
3	4	11	9
3	5	104	103
3	6	8	10
3	7	57	54
3	8	16	18
3	9	25	24
3	10	7	6
3	11	27	26
3	12	22	22

K	L	FO	FC
3	13	19	18
3	14	12	12
4	1	8	7
4	2	41	40
4	3	5	7
4	4	26	26
4	5	27	26
4	8	14	13
4	9	12	12
4	10	21	19
4	11	22	22
4	12	13	11
4	13	10	10
4	14	23	25
4	15	8	8
5	0	72	74
5	1	45	44
5	2	89	89
5	3	23	24
5	4	55	54
5	5	38	37
5	6	57	58
5	7	28	26
5	8	15	17
5	9	9	8
5	10	27	27
5	11	18	18
5	12	14	12
5	13	19	19
5	14	9	8
6	1	22	21
6	2	18	18
6	4	25	25
6	5	22	22
6	7	12	11
6	8	20	20
6	9	14	15
6	10	8	7
6	13	12	12
7	0	49	49
7	1	51	52
7	2	25	26
7	3	38	37
7	4	28	27
7	5	22	21
7	6	37	36
7	7	7	8
7	8	32	32
7	9	15	15
7	10	23	23
8	1	12	12
8	2	8	8
8	4	8	8
8	5	7	7
8	7	16	15
8	8	12	12

K	L	FO	FC
9	0	19	19
9	1	34	36
9	3	27	26
9	4	6	4

H= 14

K	L	FO	FC
0	0	121	120
0	2	116	116
0	4	121	118
0	6	102	102
0	8	53	53
0	10	12	10
0	12	15	16
0	16	15	14
1	2	29	30
1	3	11	15
1	4	12	15
1	5	6	8
1	6	35	34
1	7	29	29
1	8	50	51
1	10	5	7
1	11	12	11
1	12	31	31
1	13	28	28
1	14	57	58
1	15	23	24
1	16	26	26
2	0	44	44
2	1	49	48
2	2	51	49
2	3	75	74
2	4	23	22
2	5	74	72
2	6	56	56
2	7	44	43
2	9	45	46
2	10	14	14
2	11	11	10
2	12	29	29
2	13	9	8
2	16	10	6
3	1	21	22
3	3	26	26
3	4	17	16
3	5	24	24
3	7	50	51
3	8	33	32
3	9	25	25
3	11	32	33
3	12	14	14
3	13	39	40
3	15	26	25
4	0	19	19
4	1	72	70

K	L	FO	FC
4	2	61	60
4	3	58	59
4	4	45	45
4	5	53	54
4	7	41	41
4	8	33	31
4	9	25	26
4	10	31	31
4	11	30	30
4	12	12	14
4	13	15	15
5	3	8	9
5	4	16	15
5	5	12	13
5	6	18	17
5	7	46	47
5	8	6	5
5	9	26	25
5	10	27	27
5	12	34	34
5	13	10	10
5	14	18	17
6	0	49	50
6	2	51	52
6	3	12	12
6	4	33	34
6	5	12	12
6	6	17	16
6	7	8	9
6	8	28	26
6	10	17	18
6	12	8	7
7	2	23	23
7	3	6	8
7	6	22	21
7	8	14	15
7	10	11	10
8	0	9	8
8	1	37	37
8	2	14	15
8	3	22	22
8	4	23	21
8	5	20	19
8	6	10	10
8	7	16	15
9	1	7	7

H= 15

K	L	FO	FC
0	2	20	19
0	4	53	53
0	6	64	65
0	8	20	20
0	10	22	21
0	12	49	50
0	14	57	57

K	L	FO	FC
0	16	40	40
1	1	24	23
1	2	76	73
1	4	67	67
1	5	9	12
1	6	10	10
1	7	22	22
1	8	49	48
1	9	36	37
1	10	31	30
1	12	23	23
1	13	9	9
2	3	47	47
2	5	32	34
2	6	35	35
2	7	41	41
2	8	21	21
2	9	13	14
2	10	12	13
2	11	38	38
2	12	18	18
2	13	49	49
2	14	27	27
2	15	41	41
3	0	7	7
3	1	34	34
3	2	32	30
3	3	53	54
3	4	12	14
3	6	34	33
3	7	21	22
3	8	20	19
3	9	37	37
3	10	16	17
3	11	6	5
3	12	6	8
3	15	11	9
4	2	39	36
4	3	6	5
4	4	14	13
4	5	28	27
4	7	32	32
4	9	21	22
4	10	31	31
4	11	29	28
4	12	24	25
4	13	32	31
4	14	29	28
5	0	49	49
5	1	24	27
5	2	22	22
5	3	23	22
5	4	11	10
5	5	15	15
5	6	22	20
5	8	9	9

K	L	FO	FC
5	9	9	10
5	10	6	6
6	1	19	17
6	3	12	11
6	4	21	20
6	5	17	17
6	6	27	29
6	7	9	6
6	8	25	25
6	10	34	34
6	11	10	12
6	12	29	29
7	0	33	32
7	1	14	13
7	6	21	21
7	10	10	8
8	3	6	5
8	4	10	10
8	5	8	9
8	6	9	12
8	7	22	21

H= 16

K	L	FO	FC
0	0	32	31
0	2	26	26
0	4	16	16
0	6	13	13
0	8	21	21
0	10	29	29
1	2	5	11
1	3	40	40
1	4	60	56
1	5	35	33
1	6	52	52
1	7	16	17
1	8	6	8
1	9	10	10
1	10	45	45
1	11	32	31
1	12	45	43
1	13	32	32
1	14	34	35
1	15	24	24
2	0	43	43
2	1	37	36
2	2	16	18
2	3	17	18
2	4	64	62
2	5	31	30
2	9	10	9
2	10	8	8
2	11	10	12
2	12	6	9
2	14	6	3
2	15	7	6

K	L	FO	FC
3	2	47	48
3	3	15	13
3	5	23	22
3	7	29	29
3	9	37	38
3	10	11	14
3	11	45	44
3	13	47	46
3	14	11	13
4	0	51	49
4	1	20	19
4	3	28	26
4	4	6	7
4	5	29	28
4	6	17	16
4	7	7	4
4	8	10	11
4	10	11	12
5	1	17	18
5	2	33	35
5	3	18	18
5	4	7	5
5	6	28	26
5	8	45	47
5	9	17	17
5	10	36	35
5	11	17	16
5	12	28	27
6	0	12	12
6	1	25	24
6	3	10	6
6	5	12	10
6	6	25	24
6	8	10	9
6	9	15	14
6	11	7	5
7	2	12	10
7	3	13	12
7	5	9	10
7	6	14	16
7	7	17	17
7	8	35	35
7	9	15	16
8	1	7	9
8	3	8	8

H= 17

K	L	FO	FC
0	2	91	89
0	4	32	33
0	6	10	10
0	8	41	40
0	10	26	25
0	12	36	37
0	14	40	40
1	0	80	79

K	L	FO	FC
1	1	72	70
1	2	76	75
1	3	20	19
1	4	58	57
1	5	27	28
1	6	42	42
1	7	18	19
1	10	11	7
1	11	11	9
1	12	18	19
1	13	7	6
2	1	12	18
2	2	37	37
2	3	54	55
2	4	13	13
2	5	39	40
2	6	13	14
2	7	20	20
2	8	51	51
2	9	31	30
2	10	22	21
2	11	38	39
2	12	12	11
2	13	39	38
2	14	20	20
3	0	23	25
3	1	56	56
3	3	46	45
3	4	38	41
3	5	53	55
3	6	11	13
3	7	22	21
3	8	6	6
3	9	24	24
3	11	19	19
3	13	18	18
4	2	11	11
4	4	51	52
4	6	34	36
4	7	28	28
4	8	12	12
4	9	41	39
4	10	19	19
4	11	24	25
4	12	16	18
4	13	23	21
5	0	46	47
5	1	12	10
5	2	27	28
5	3	23	25
5	4	24	22
5	5	16	18
5	6	32	32
5	7	12	11
5	8	11	12
5	10	15	12

K	L	FO	FC
5	11	9	9
5	12	7	8
6	1	10	9
6	2	10	8
6	3	10	6
6	4	23	23
6	5	29	28
6	6	26	27
6	8	25	24
6	10	37	36
7	0	30	28
7	1	11	12
7	2	31	32
7	3	7	4
7	4	20	20
7	5	9	10
7	6	16	16
7	7	8	7
8	2	7	5

H= 18

K	L	FO	FC
0	0	113	105
0	2	75	78
0	4	44	47
0	6	103	100
0	8	21	21
0	10	25	26
0	12	38	37
1	3	58	60
1	4	62	62
1	5	34	33
1	8	8	8
1	9	16	15
1	10	36	36
1	11	14	13
1	12	22	23
2	0	82	81
2	1	66	64
2	2	28	30
2	3	46	49
2	4	23	22
2	5	66	66
2	6	41	40
2	7	46	45
2	8	16	18
2	9	26	27
2	11	29	29
2	12	12	13
2	13	18	20
3	2	26	25
3	3	29	29
3	4	13	13
3	5	46	46
3	6	7	6
3	9	24	24

K	L	FO	FC
3	10	18	19
3	11	27	27
3	12	7	9
3	13	11	11
4	0	23	22
4	1	51	52
4	2	35	34
4	3	46	46
4	4	34	35
4	5	44	44
4	6	12	11
4	7	33	33
4	8	17	17
4	9	31	31
4	10	18	19
4	11	16	16
5	4	11	12
5	5	8	6
5	6	11	12
5	7	14	14
5	8	13	12
5	9	15	16
5	10	11	12
5	11	12	11
6	0	27	26
6	2	52	52
6	3	8	3
6	4	47	48
6	6	21	21
6	8	25	24
7	6	9	7

H= 19

K	L	FO	FC
0	4	23	22
0	6	32	34
0	8	22	21
1	0	22	23
1	1	17	21
1	2	67	66
1	3	44	42
1	4	61	62
1	5	36	36
1	6	25	27
1	7	21	21
1	8	54	53
1	9	15	15
1	10	49	48
1	11	21	21
1	12	11	11
1	13	6	7
2	3	29	29
2	4	8	7
2	5	19	20
2	7	17	17
2	10	19	17

K	L	FO	FC
3	0	15	15
3	1	65	66
3	3	54	53
3	5	51	49
3	6	29	30
3	7	44	45
3	8	16	15
3	9	49	49
3	10	7	10
3	11	23	23
3	12	16	16
4	2	7	9
4	4	11	11
4	6	7	9
4	8	13	10
4	9	14	12
4	10	8	7
4	11	6	2
5	0	59	58
5	1	21	21
5	2	33	32
5	3	35	35
5	4	31	31
5	5	8	7
5	6	43	44
5	7	11	10
5	8	35	35
5	9	22	21
5	10	19	18
6	4	7	7
6	5	10	8
6	6	7	8
6	7	12	11
7	0	23	23
7	1	23	21
7	2	28	28
7	3	17	16
7	4	24	24
7	5	20	19

H= 20

K	L	FO	FC
0	0	67	69
0	2	42	45
0	4	31	31
0	6	62	64
0	8	54	54
0	10	23	22
0	12	15	16
1	4	20	18
1	5	12	14
1	6	27	27
1	7	16	14
1	8	18	18
1	9	20	22
1	10	36	37

K	L	FO	FC
1	11	12	12
1	12	19	17
1	13	10	8
2	0	43	43
2	1	48	47
2	3	38	38
2	5	33	34
2	6	23	24
2	7	41	40
2	8	32	31
2	9	24	26
2	10	11	6
2	11	16	16
2	12	7	10
3	2	16	19
3	3	12	16
3	4	30	30
3	5	28	28
3	7	40	40
3	8	24	25
3	9	18	20
3	11	17	17
4	0	19	20
4	1	50	50
4	2	24	24
4	3	32	32
4	4	34	32
4	5	26	28
4	6	15	13
4	7	33	32
4	8	19	19
4	9	22	24
4	10	16	15
4	11	6	6
5	2	11	11
5	3	13	12
5	5	21	21
5	6	28	28
5	8	25	27
6	0	20	18
6	2	33	33
6	3	9	8
6	4	32	31
6	6	18	17
7	1	7	4
7	2	9	8
	H=	21	
0	2	46	53
0	4	6	5
0	6	31	31
0	8	67	66
0	10	39	40
0	12	25	24
1	0	56	56

K	L	FO	FC
1	1	20	20
1	2	35	37
1	4	19	21
1	6	22	21
1	7	22	22
1	8	20	21
1	9	9	7
2	2	26	27
2	3	15	16
2	5	20	21
2	7	48	46
2	8	22	22
2	9	42	41
2	10	10	8
2	11	22	22
2	12	12	12
3	1	39	39
3	3	39	39
3	5	13	15
3	7	13	13
3	8	7	10
3	9	14	14
4	1	8	10
4	2	16	18
4	3	27	28
4	4	8	8
4	5	26	26
4	6	25	25
4	7	25	27
4	8	15	15
4	9	19	19
4	10	12	14
5	0	18	18
5	1	18	20
5	2	21	21
5	3	16	16
5	4	24	25
5	5	8	7
5	8	13	14
6	1	12	11
6	3	9	11
6	4	24	23
	H=	22	
0	0	9	16
0	2	29	35
0	4	21	22
0	6	13	11
1	3	12	12
1	4	8	11
1	5	13	13
1	6	23	23
1	7	19	19
1	8	27	26
1	9	24	24

K	L	FO	FC
1	10	36	36
1	11	19	18
2	0	14	14
2	4	26	26
2	5	9	10
2	6	22	20
3	2	7	6
3	4	10	13
3	5	23	21
3	7	27	28
3	8	13	12
3	9	36	36
4	0	11	14
4	3	13	12
5	2	11	11
5	3	9	11
5	4	12	13
5	5	12	10
5	6	15	15
5	7	8	8
6	0	15	16
	H=	23	
0	4	14	11
0	6	20	20
0	8	25	25
0	10	30	31
1	0	12	11
1	1	29	29
1	2	23	25
1	3	12	13
1	4	20	21
1	5	13	12
1	8	15	15
1	10	15	16
2	4	17	16
2	5	6	8
2	7	16	16
2	9	30	30
3	0	16	17
3	1	23	23
3	2	7	7
3	3	21	22
3	4	6	9
3	5	17	17
3	6	8	6
3	7	12	13
3	9	15	16
4	2	10	8
4	3	11	13
4	5	7	4
5	0	14	13
5	1	6	6
5	2	8	8
5	3	13	13

K	L	FO	FC
5	4	8	9
	H=	24	
0	0	69	72
0	2	39	41
0	4	37	35
0	6	32	32
0	8	28	29
1	6	7	5
1	7	8	8
1	8	26	26
1	9	18	17
2	0	24	26
2	1	47	47
2	2	30	30
2	3	30	30
2	4	23	24
2	5	31	30
2	6	20	20
2	7	26	26
2	8	11	10
3	2	11	11
3	6	7	4
4	0	14	13
4	1	28	29
4	2	19	18
4	3	26	26
4	4	16	15
4	5	26	25
	H=	25	
1	0	57	57
1	1	33	33
1	2	42	44
1	3	26	26
1	4	39	39
1	5	17	17
1	6	32	31
1	7	10	9
2	4	15	17
3	1	35	35
3	3	41	40
3	5	34	35
	H=	26	
0	0	35	35
0	2	41	40
0	4	42	43
0	6	9	9
1	4	7	10
2	1	35	34
2	2	18	18
2	3	34	33

K	L	FO	FC
2	4	16	19
2	5	19	19

H= 27

1	0	27	29
1	1	14	15
1	2	11	12

Cu(L2am)(ClO_4)

K	L	FO	FC	K	L	FO	FC	K	L	FO	FC	K	L	FO	FC
	H=-13			6	5	12	13	5	1	9	9	1	4	24	24
				6	6	23	23	5	3	22	22	1	5	5	5
4	2	9	9	6	7	11	10	5	4	21	21	1	6	4	6
4	3	8	8	6	9	6	5	5	5	27	28	1	8	6	6
4	5	6	7	7	1	11	11	5	6	12	12	1	10	6	5
4	6	5	5	7	2	8	9	5	8	4	4	1	11	9	9
5	1	13	11	7	3	8	9	6	1	12	12	2	1	22	21
5	2	19	19	7	4	9	10	6	2	3	1	2	2	9	8
5	3	15	15	7	5	15	16	6	3	14	13	2	3	21	21
5	5	3	4	7	6	20	21	6	4	9	9	2	4	23	23
6	1	12	12	7	8	8	7	6	5	13	13	2	6	8	8
6	2	18	18	8	1	4	3	6	6	6	6	2	7	10	10
6	3	4	4	8	3	8	8	6	7	9	8	2	8	12	12
7	1	6	5	8	4	11	10	6	10	4	3	2	9	14	14
7	2	14	13	8	5	12	12	7	1	4	6	2	12	12	12
7	3	6	4	8	6	4	3	7	2	7	8	3	1	16	15
7	4	5	4	9	4	9	9	7	3	7	7	3	2	18	19
8	1	10	11	9	5	10	11	7	4	27	27	3	3	20	20
8	2	22	22	9	6	13	12	7	5	21	21	3	4	29	28
8	3	16	17	10	3	6	5	7	6	11	11	3	5	16	16
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K	L	FO	FC
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-7	11	10	9
-6	1	21	21
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-6	3	7	8
-6	5	7	7
-6	6	8	8
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3	3	6	7
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3	6	11	11
4	0	13	13
4	1	8	7
4	3	11	11
4	5	9	9

H= 7

K	L	FO	FC
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-9	6	10	8
-9	7	8	9
-8	0	22	23
-8	2	16	16
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-8	8	10	9
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-1	7	13	14
0	0	6	6
0	1	6	6
0	2	6	7
1	1	15	16
1	3	9	10

K	L	FO	FC
2	0	7	5
2	2	11	11

H= 8

K	L	FO	FC
-6	1	8	9
-5	2	7	6
-4	1	9	10
-3	0	11	10

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